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WEAR RESISTANT COATINGS FOR TITANIUM.(U)

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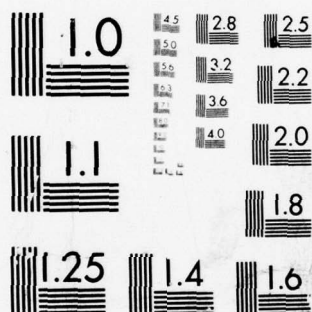
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WEAR RESISTANT COATINGS FOR TITAN

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Pratt & Whitney Aircraft Group
Government Products Division
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JUNE 1978

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PREFACE

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SECTION I

INTRODUCTION

This report describes the results of the second year of a planned 4-year program to develop wear-resistant chromium-molybdenum coatings and apply these coatings to candidate titanium alloy components. The capability to use titanium for gas turbine or airframe components often depends on a suitable surface treatment being available to alleviate inherent problems such as wear, erosion, and corrosion.

The first year of this effort established that the chromium-molybdenum system can be applied to titanium alloys to produce a wear-resistant surface without attendant reduction of mechanical properties. Further, excellent oxidation and erosion resistance was obtained using this coating on titanium.

During this second program phase, an investigation was undertaken to define the extent to which the chromium-molybdenum coatings, developed in Phase I, influenced the fatigue strength of Ti-8Al-1V-1Mo and Ti-6Al-4V. Both notched fatigue and smooth fatigue tests were run using a temperature range from room temperature to 590°C (1100°F). Creep tests were conducted on Ti-8Al-1V-1Mo, and Ti-6Al-2Sn-4Zr-2Mo. Tensile tests were conducted on Ti-6Al-4V to assess the influence of various coating process modifications on the creep, yield strength, tensile strength, and elongation.

Included in this second phase was a preliminary study of the effects of pulse-plating on the deposits obtained. Surface morphology, composition, hardness, wear resistance, and deposit cracking were all investigated as a function of the pulse-current duration. The effect of heat treat on the hardness of pulse plated and direct-current plated samples was measured up to a temperature of 960°C (1800°F).

In addition, oxidation tests at temperatures up to 927°C (1700°F) were conducted plus additional wear tests to further characterize the coating systems.

The problems associated with scale-up and plating of a large complex geometry gas turbine engine part were studied using a compressor sync ring. The completion of this second phase has:

1. Provided additional information on the properties of chromium-molybdenum coatings applied to titanium
2. Demonstrated the potential use of the coating on steels
3. Improved the coating quality via process modifications
4. Shown scale-up to actual hardware can be accomplished with minimal problems.

SECTION II

EXPERIMENTAL PROGRAM

The experimental portion of this contract contained the following:

1. Improved the procedure to deposit a chromium-molybdenum coating onto titanium alloys
2. Determined the amount of hydrogen and oxygen picked up after each step of the plating process
3. Evaluated the hardness of the coating
4. Evaluated the effects of the coating process on
 - Tensile strength, yield strength, and elongation
 - High-cycle fatigue
 - Creep
5. Determined the effect of the coating on the oxidation properties
6. Evaluated the wear resistance
7. Plate complex engine parts.

The object of the second phase of this program was to improve the qualities of the selected coating process and evaluate the effects that the coating has on mechanical properties.

MATERIALS

Chemical compositions of the substrate titanium alloy materials used in this investigation are listed in Table 1. These compositions were all confirmed by Pratt & Whitney Aircraft.

TABLE 1. CHEMICAL COMPOSITION OF TITANIUM ALLOY SUBSTRATE MATERIALS

Alloy	Test	Heat Code	Composition, Weight (%)							
			Al	Mo	V	Fe	N	C	O	H
Ti 8Al-1Mo-1V	Fatigue	SGRZ	7.3	0.94	0.85	0.15	0.015	0.015	—	0.009
	Creep	SGRZ	7.3	0.94	0.85	0.15	0.015	0.015	—	0.009
	Tensile	SGRZ	7.3	0.94	0.85	0.15	0.015	0.015	—	0.009
	Oxidation	SGRZ	7.3	0.94	0.85	0.15	0.015	0.015	—	0.009
	Hydrogen	SGRZ	7.3	0.94	0.85	0.15	0.015	0.015	—	0.009
	Oxygen	SGRZ	7.3	0.94	0.85	0.15	0.015	0.015	—	0.009
Ti 6Al-4V	Fatigue	BDHB	6.55	—	4.3	0.22	0.016	0.023	0.20	0.009
	Creep	BDHB	6.55	—	4.3	0.22	0.016	0.023	0.20	0.009
	Hydrogen	BDHB	6.55	—	4.3	0.22	0.016	0.023	0.20	0.009
	Oxygen	BDHB	6.55	—	4.3	0.22	0.016	0.023	0.20	0.009

Chemicals used in preparing the plating solutions and other solutions used in this investigation were all reagent grade materials except for the self-regulating chromic acid which was obtained from United Chromium Corporation under their designation SRHS CR 110.

COATING APPLICATION PROCEDURE

Test Specimen Coating

Coating of all the mechanical test specimens was accomplished using the techniques developed in Phase 1 as reported in Reference 1.

This procedure consisted of preparing the surfaces for plating by vapor blasting with Novaculite 200 (aluminum oxide) abrasive at 0.35 to 0.7 MPa (50 to 100 psig) pressure followed by a chromium conversion coating. The conversion coating was applied by immersing the titanium specimen in a solution containing hydrofluoric acid and sodium chromate. After preparation, the specimens were placed in a bath containing 300 g/l self-regulating chromic acid and 75 g/l ammonium molybdate. Plating was accomplished at 46.7ASD for 30 min. The specimens were then diffusion heat treated in vacuum at 760°C (1400°F) for 3 hr. Finally, the surfaces were lightly vapor blasted to remove any oxidation occurring during the diffusion cycle. This procedure is further detailed in Appendix A.

Pulse-Plating Development

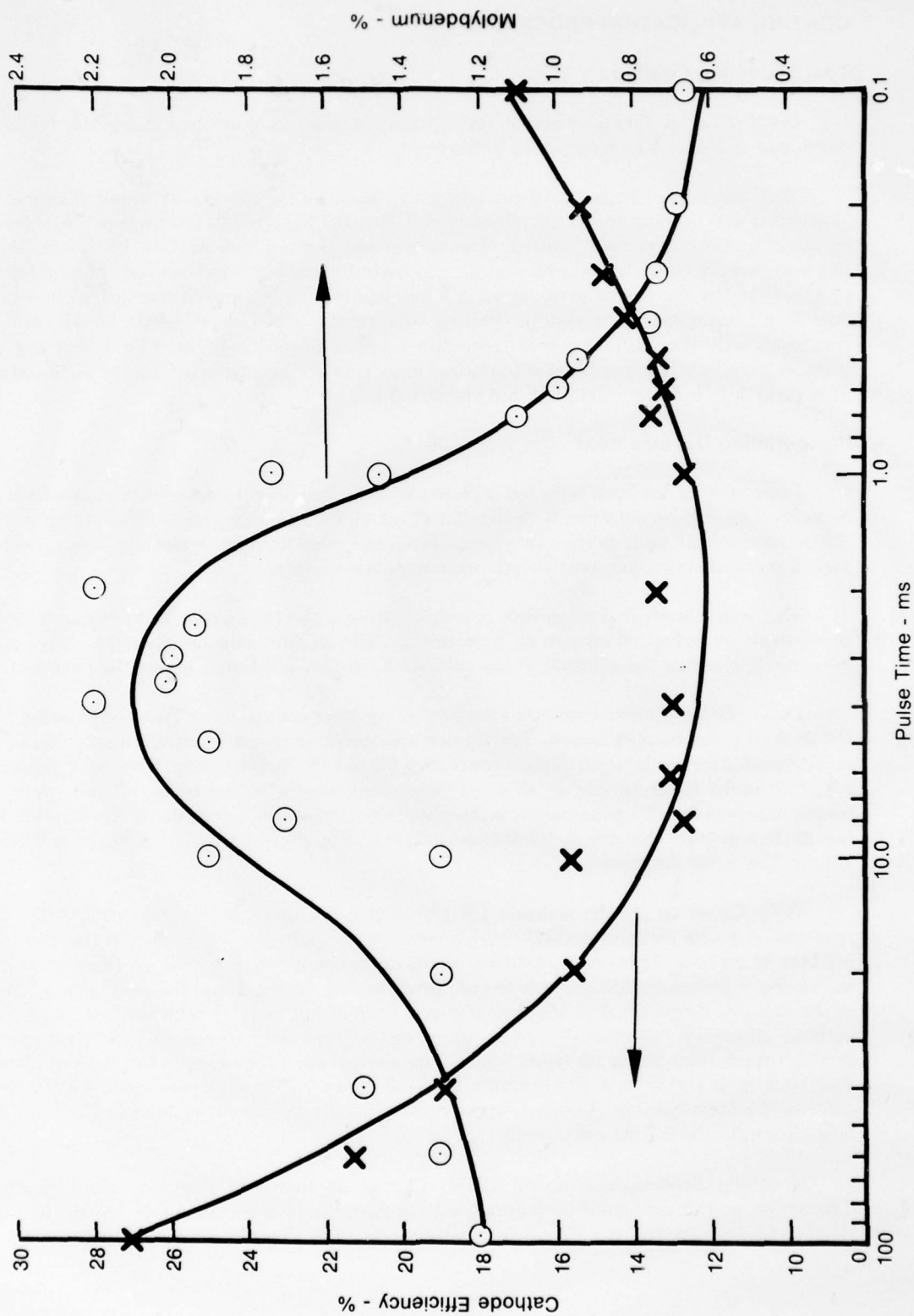
Pulse-plating has been reported (References 2 through 6) to produce less nodular, finer grain deposits than can be obtained with direct current alone. It accomplishes this without reducing the deposition rate and, at the same time, minimizes edge buildup or treeing. This gives better overall metal distribution, particularly on a large flat surface.

The main theoretical advantage of pulse-plating is the elimination of hydrogen bubbles at the cathode by reduction of cathode polarization. The off-time pulse is, therefore, more critical in terms of allowing the solution at the cathode to reach equilibrium before the next on pulse.

Pulse-plating studies conducted under this contract used a Nova Tran Corporation Model DP-20-5-10 pulse current source. Twenty-one specimens were coated using the plating solution recommended for producing a deposit containing 0.5 to 1.0% molybdenum. The on/off pulses were varied from 0.1 to 99 ms and in all cases the on-time was set equal to the off-time pulse. The results obtained for a 75 g/l ammonium molybdate electrolyte are shown in Figures 1 and 2. These results demonstrate that the molybdenum content can be controlled over a wide range by simply varying the pulse duration.

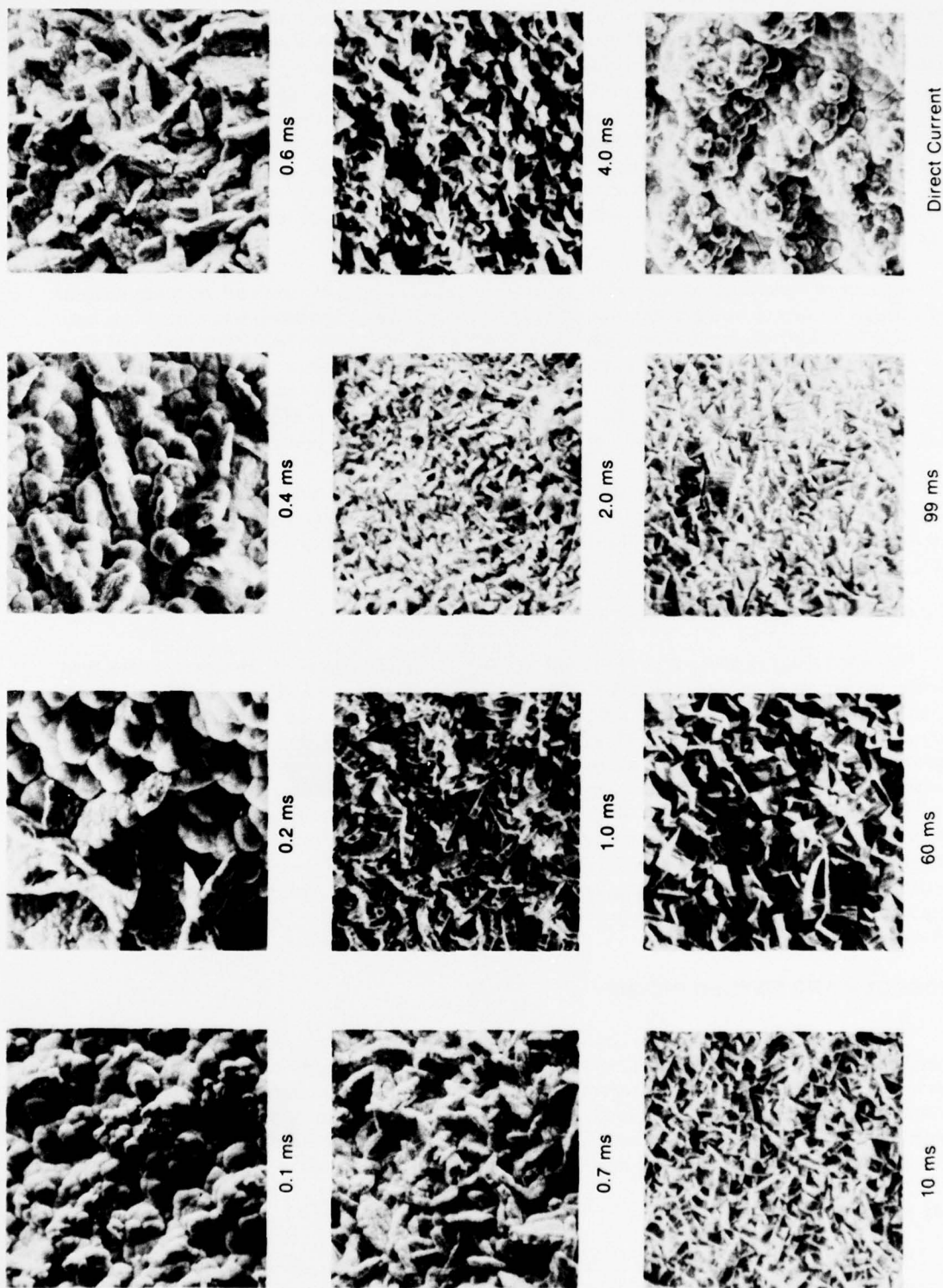
With the same plating solution (75 g/l ammonium molybdate) conventional dc plating produced deposits containing 0.42% molybdenum while pulse plating increased this amount by a factor of up to 5. This effect occurred at the optimum pulse time of 3 ms (Figure 1). Either increasing or decreasing the pulse from the optimum 3 ms time reduced the molybdenum content of the deposit. As reported in the first phase, a decrease in molybdenum content increases the cathode efficiency. Interestingly, pulse times below 3 ms reduced the cathode efficiency to a greater extent than did pulse times above 3 ms even though they resulted in the same percent molybdenum in the deposit. For example, both a 99 and a 0.7 ms pulse resulted in a molybdenum content of a little over 1%. However, the cathode efficiency for the 99 ms pulse (~25%) was much larger than for the 0.7 ms pulse (~13%).

Scanning electron microscopy (SEM) photos, as shown in Figure 2, show significant differences in the topography of deposited chromium-molybdenum alloys using the 75 g/l electrolyte. In every test utilizing pulse-plating, crack-free deposits were obtained.



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Figure 1. Influence of Pulse Time on Molybdenum Content of Deposit and Cathode Efficiency



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Figure 2. Pulse Plated Specimens Using Electrolyte Containing 75 g/l Ammonium Molybdate

Using an electrolyte containing 95 g/l ammonium molybdate, additional studies of cathode efficiency and molybdenum content were conducted. As before, on- and off-plating periods, of equal duration, were varied over the range of 0.1 to 99 ms. The results obtained, as shown on Figures 3 and 4, reinforced the premise that molybdenum content is controllable over a wide range by variation of pulse duration. SEM photos (Figure 3) show a surface topography similar to that obtained with the 75 g/l ammonium molybdate plating solution. With increasing on/off-pulse duration, topography of coated surfaces ranged from fine structured nodules to randomly oriented rods, and rectangularly shaped crystals. Fine nodule covered, unevenly spaced, rounded protuberances are seen in the direct-current-plated photo. The effect these structures would have on wear properties may be significant. The rods or rectangles may prove ideal for lubricant retention.

A series of tests were conducted to explore the relationship between molybdenum content and cathode efficiency at a constant on/off pulse cycle of 3 ms. A specimen was plated in a bath consisting of 75 g/l of ammonium molybdate and 300 g/l of chromic acid. The bath was then modified by the addition of 10 g ammonium molybdate and a second specimen plated. This procedure was repeated four more times. Only slight differences in topography were observed after each addition as shown in Figure 5. Figure 6 illustrates the effect of the ammonium molybdate concentration on the cathode efficiency and percent molybdenum in the deposit.

To illustrate the crack-free nature of the deposits produced by pulse-plating, the specimens shown previously in Figure 1 were electropolished with the results seen in Figure 7. These electropolished specimens show strikingly different results from sample to sample. It should be noted that at long on/off pulse durations the deposits are no longer crack-free.

Post-Heat-Treat Surface Cleaning

Post-heat-treat cleaning is not required as a functional consideration. However, unless heat treating is performed in a completely oxygen-free environment, some surface discoloration will occur. Since a uniformly clean, matte finish is desirable to facilitate inspection of the final coating, tests were conducted to evaluate various cleaning processes. Figure 8 shows the surface appearance after vapor blasting, glass bead peening, glass bead peening followed by vapor blasting, and vapor blasting followed by glass bead peening. Vapor blasting produced the best, most reproducible surface finish. Glass bead peening alone generally left nonuniform surface discoloration while glass bead peening followed by vapor blasting showed some indications of chipping of the coating at the specimen edges. Vapor blasting followed by glass bead peening seemed to remove the most coating and also left a poor finish. Based on these results the remainder of the specimens coated for this program were vapor blasted to effect a reproducible and clean surface.

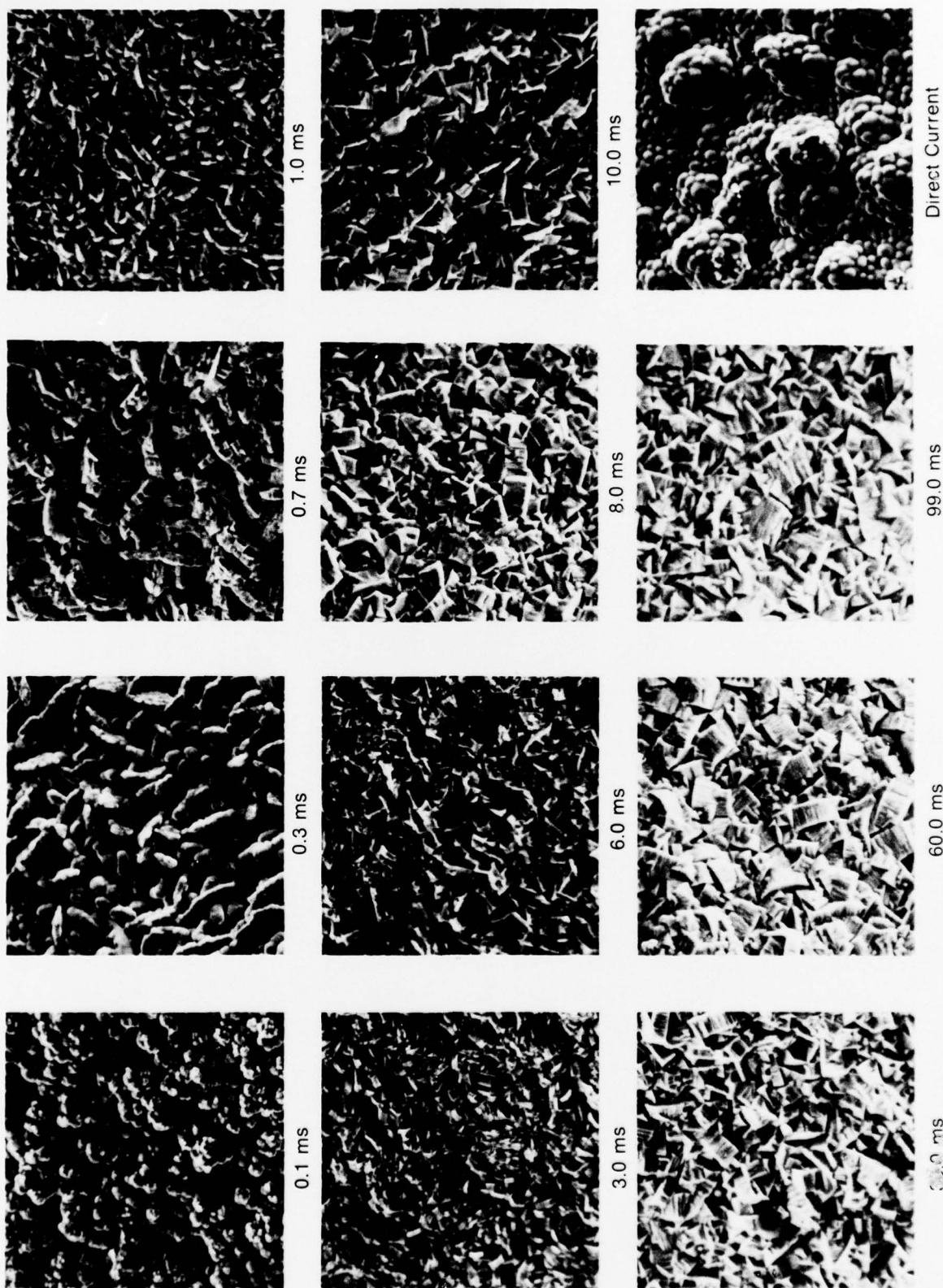
HYDROGEN AND OXYGEN PICKUP

This part of the program was conducted to determine the extent of hydrogen and oxygen pickup resulting from the coating process. Titanium alloys are severely embrittled by hydrogen absorption when exposed to a hydrogen-containing environment. During cathodic treatment in chromic acid, approximately 90% of the total current is used for generation of hydrogen. The oxygen content of the base system can be increased during different steps in the coating process but especially during the diffusion heat treat.

Test Method

Ti 6Al-4V and Ti 8Al-1Mo-1V hydrogen and oxygen absorption specimens (2 by 2 in.) were fabricated and then scored, in checkerboard fashion to produce a large number of 1/4-in. squares interconnected by thin metal webs. These specimens were coated with chromium-molybdenum and 4 squares removed after each step of the coating process.

Numbers Denote On/Off Time of Pulse



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Figure 3. Pulse Plated Chromium-Molybdenum Surfaces Using Electrolyte Containing 95 g/l Ammonium-Molybdate

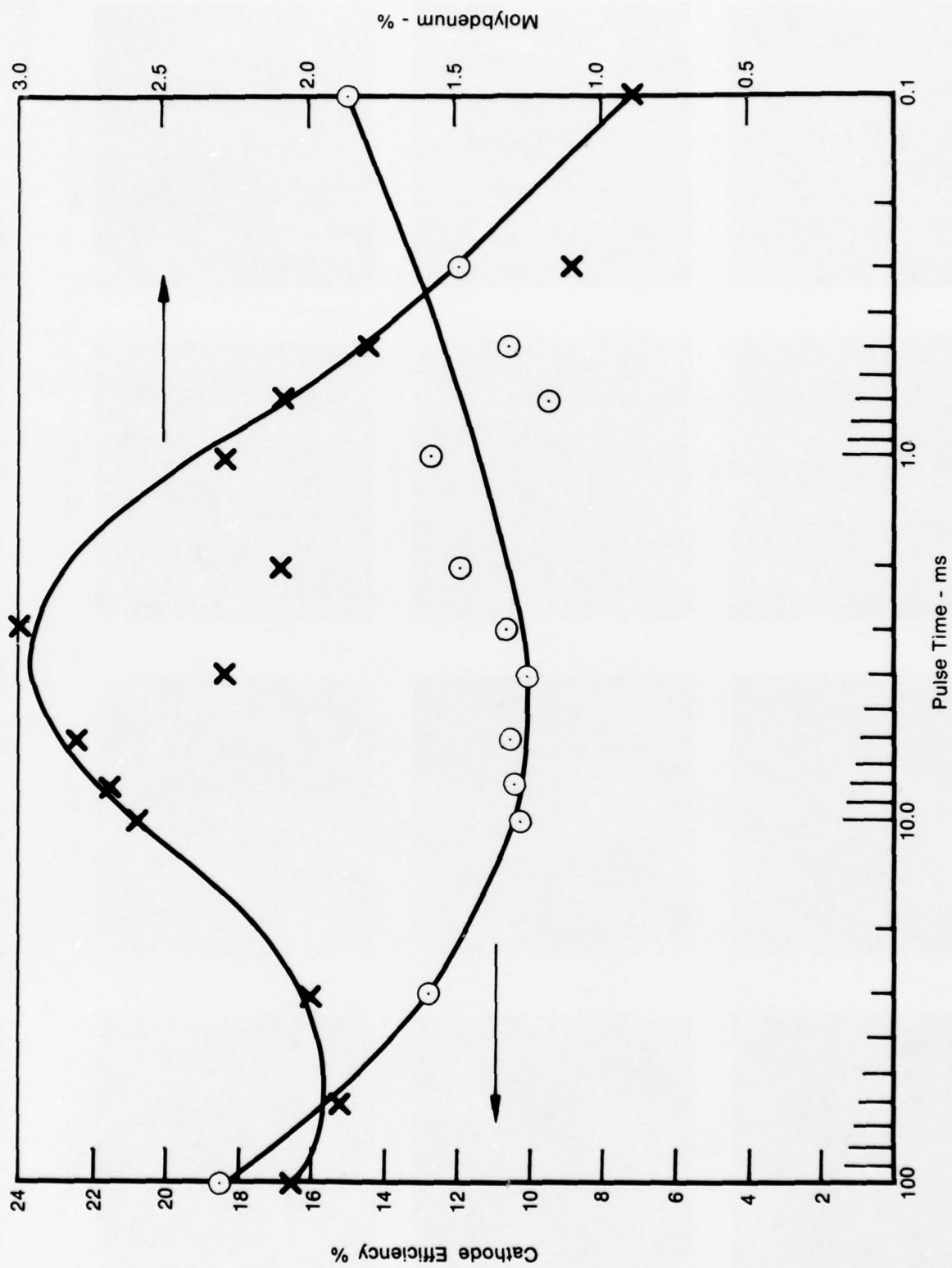


Figure 4. Influence of Pulse Time on Molybdenum Content of Deposit and Cathode Efficiency Using a Plating Solution Containing 85 g/l Ammonium Molybdate

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Numbers Denote Ammonium-Molybdate Concentration



75 g/l



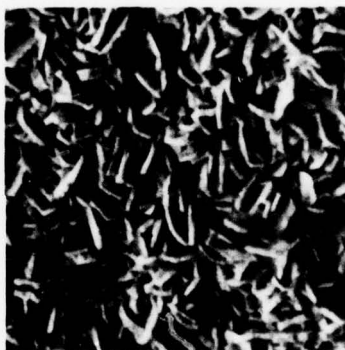
85 g/l



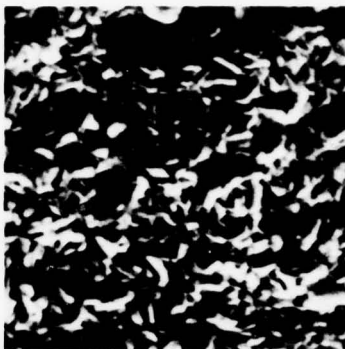
95 g/l



105 g/l



115 g/l

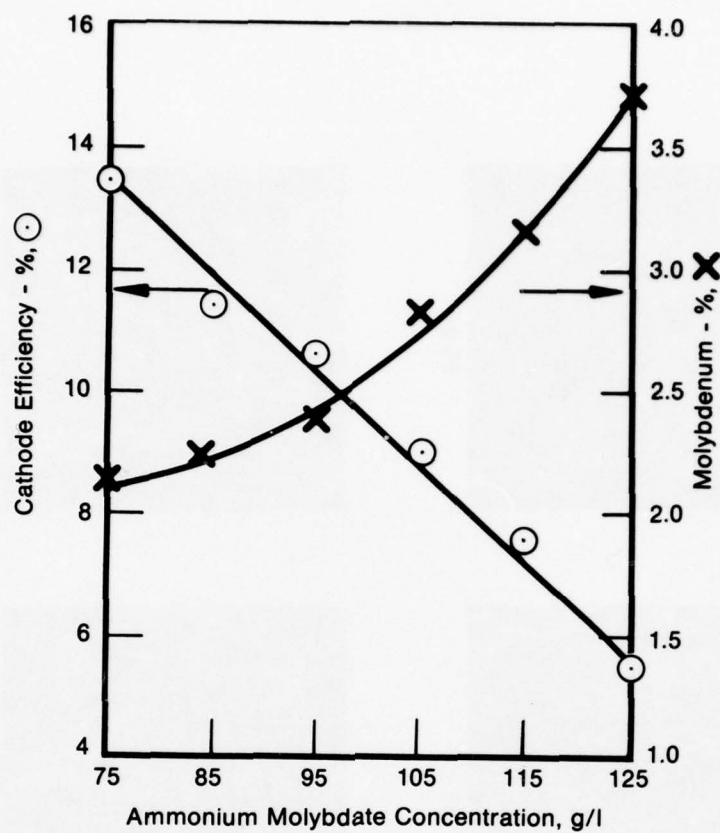


125 g/l

Mag: 1000X

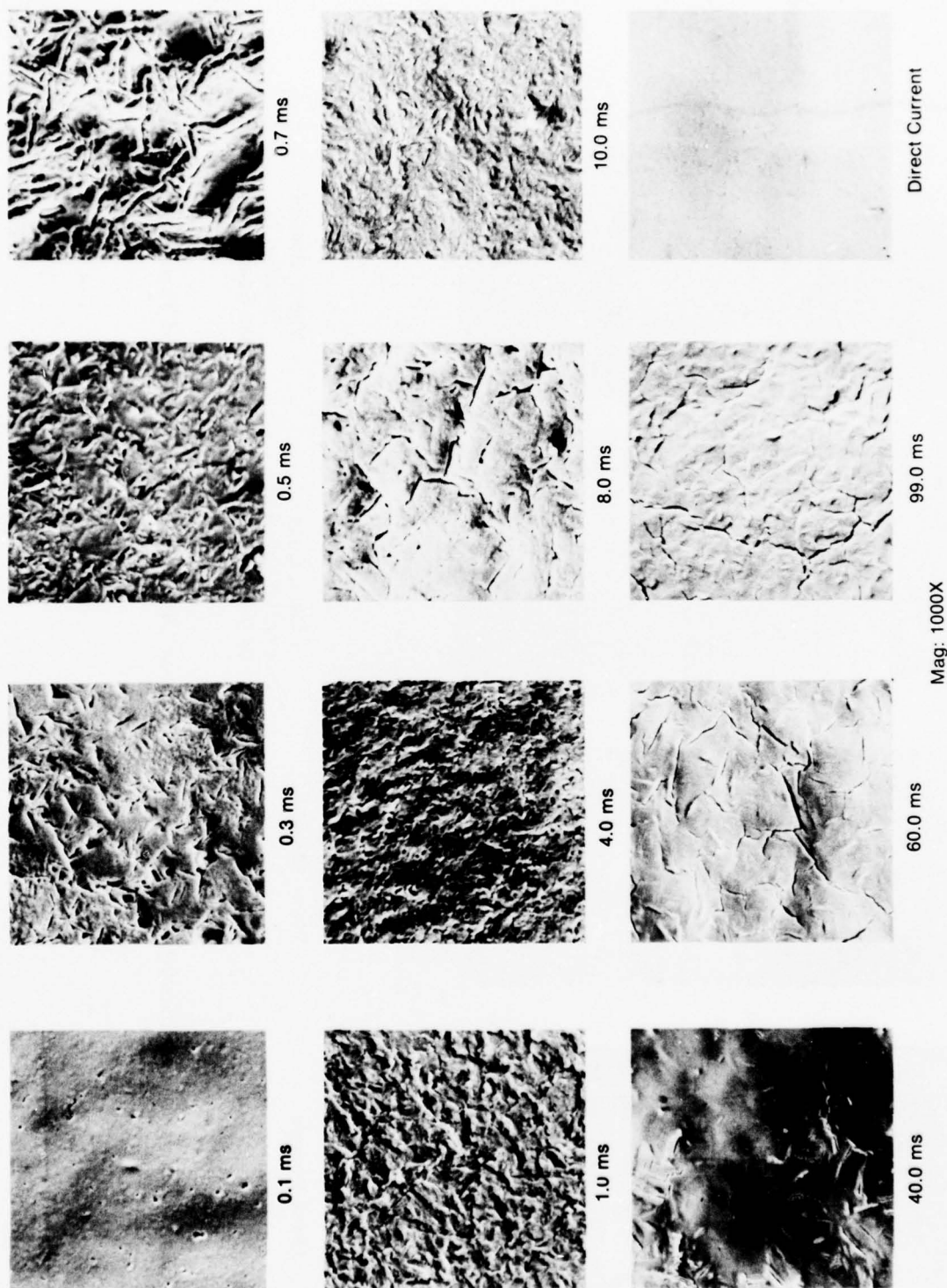
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Figure 5. Pulse Plated Chromium-Molybdenum Surfaces Plated With 3 ms Current Pulse Current



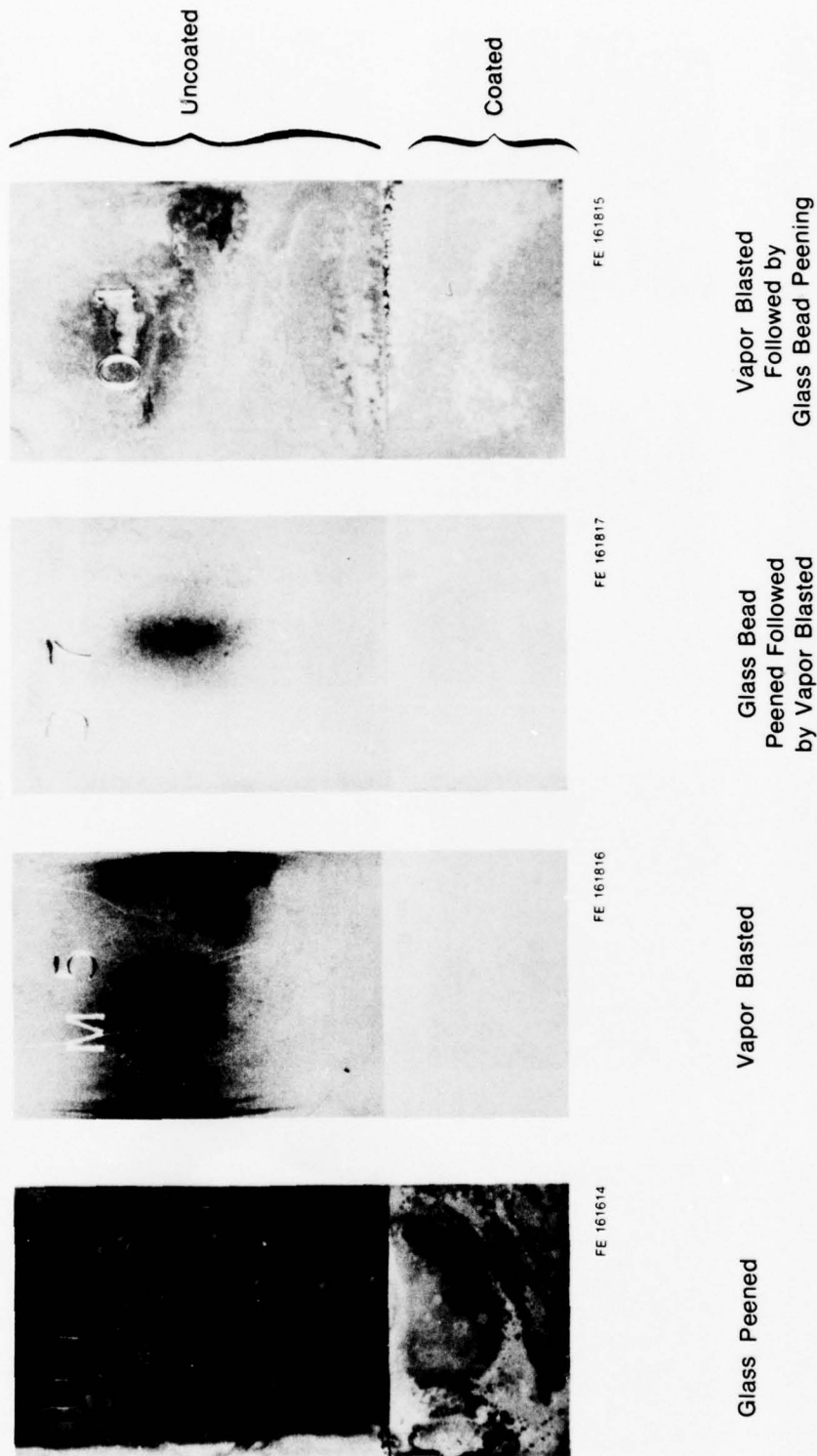
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Figure 6. The Effect of Ammonium Molybdate Concentration on Cathode Efficiency and the Percent Molybdenum Deposited



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Figure 7. Electropolished Pulse Plated Specimens Using 1% Chromium-Molybdenum Electrolyte

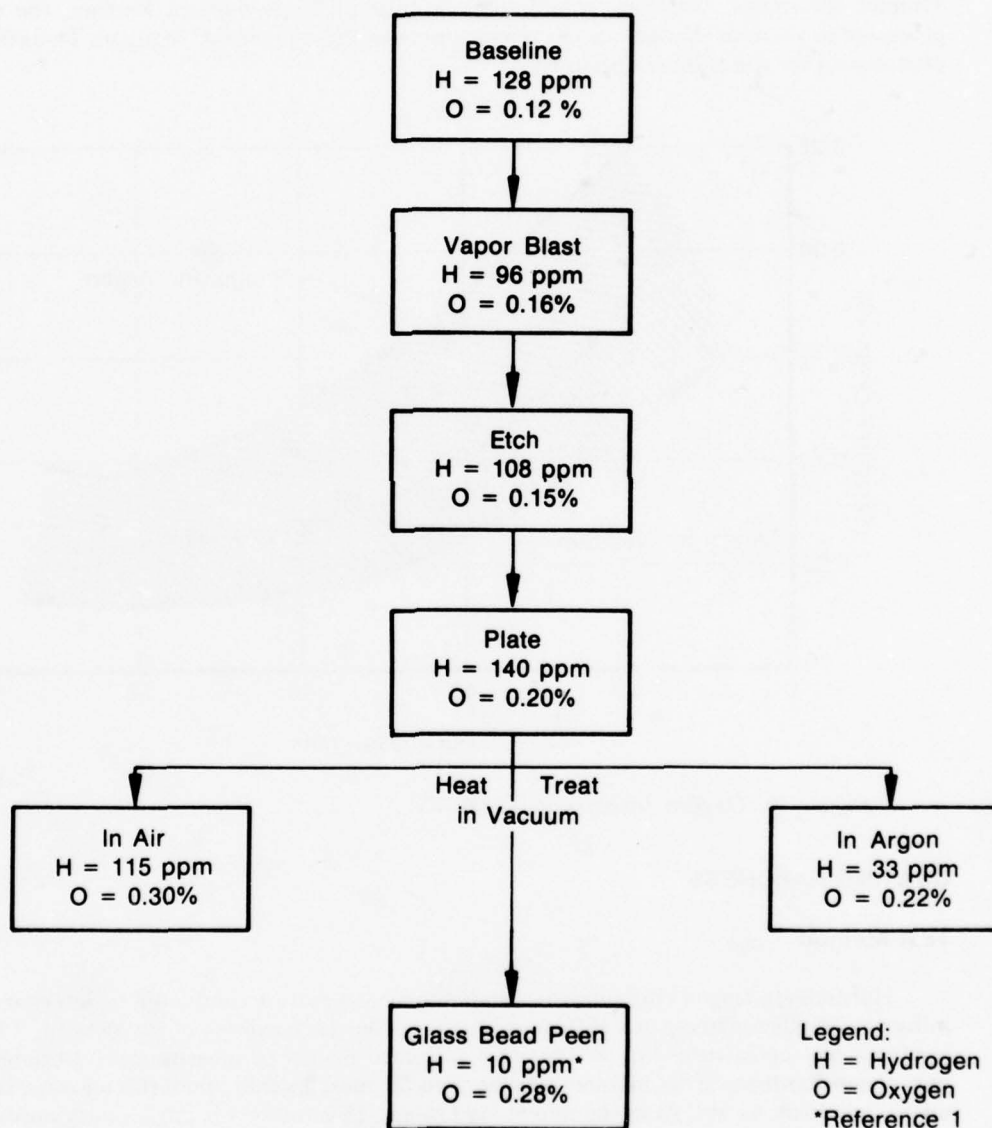


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Figure 8. Effects of Vapor Blasting and/or Glass Bead Peening on Chromium-Molybdenum Coated Titanium Specimens

Results

The concentration of hydrogen and oxygen in Ti-8Al-1V-1Mo after each step of the coating process is shown in Figure 9. Vapor blasting increased the oxygen concentration slightly, probably from imbedded aluminum oxide abrasive. During the etching cycle, some of the aluminum oxide was removed so the oxygen concentration decreased. Plating again caused an increase in oxygen content, probably the result of a small amount of oxides occluded in the deposit. When the diffusion heat treat was conducted in air, the largest oxygen pickup occurred; however, in an argon environment, this pickup seemed to be minimized. In all cases, the measured oxygen concentration exceeded the specification limit of 0.12%. Since the material used for this part of the study had an initial oxygen content of the maximum allowed, these results were expected.

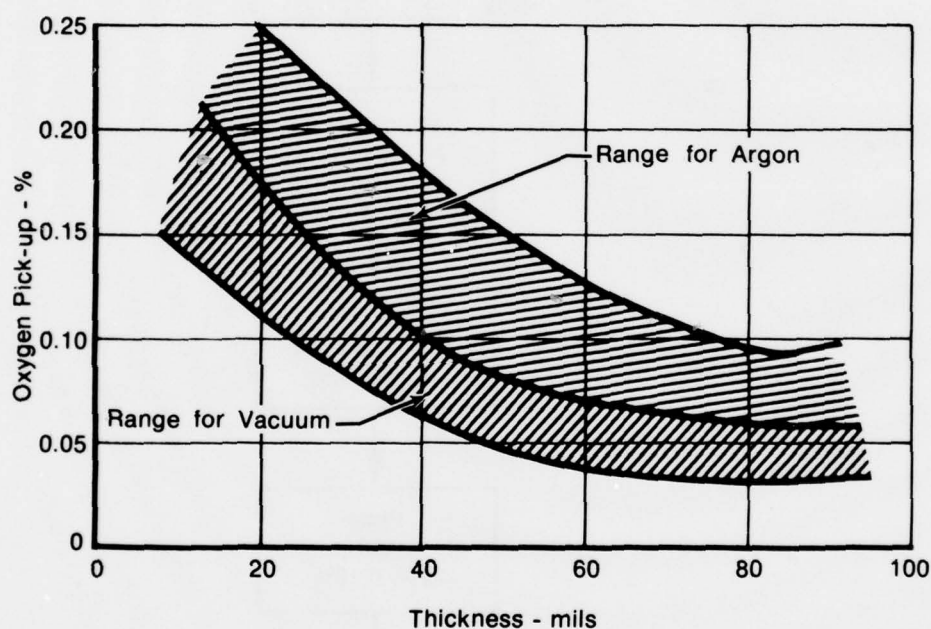


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Figure 9. Oxygen and Hydrogen Increase in Ti-8Al-1Mo-1V(Thickness: 0.038 in.)

The largest hydrogen concentration increase occurred during plating, but never exceeded specification limits. In fact, after the diffusion heat treat in argon, the hydrogen concentration was lower than the as-received material.

The relationship between different thicknesses of Ti-6Al-4V (AMS 4911) specimens and the amount of oxygen picked up during the coating procedure was studied. Oxygen analysis, both before and after processing, was used to generate the curves shown in Figure 10. Heat treating was done at 760°C (1400°F) for 3 hr in either vacuum (10 to 15 torr) or industrial grade argon. Using this analysis, the oxygen concentration increase in titanium can be predicted based on the thickness of the titanium alloy sheet. The thicker the sheet, the less the percent of oxygen increase. This thickness effect is the result of, and is exponentially related to, geometry effects. That is, oxygen content near the surface is much greater than that in the bulk of the material. Thinner specimens, therefore, would seem to pick up more oxygen. Further, the specimens processed in vacuum showed less oxygen pickup than those processed in argon. This effect is more pronounced for the thinner specimens.



FD 131866

Figure 10. Oxygen Increase in Ti 6Al-4V

COATING HARDNESS

Test Method

Hardness testing of chromium-molybdenum coatings were conducted to determine both the influence of pulse-plating and diffusion heat treat on the hardness of the deposit. The Vickers hardness test constitutes one of the most accurate means of measurement to determine the impression-hardness. This method may be used for most metallic material allowing tests in the micro- low-load, as well as in the macro-load range. This method is particularly appropriate for tests on very small, hard, and thin specimens as well as for the hardness determination on coatings.

The penetrator used is a four-faced, square (tetragonal) diamond pyramid meeting at an angle point of 136 deg between opposite faces. The tip of the penetrator is not off-rounded and even under microscopic examination appears as a point. To determine the hardness according to Vickers, the diamond pyramid is impressed at right angles (perpendicularly) into the polished surface of a specimen at a predetermined load-step.

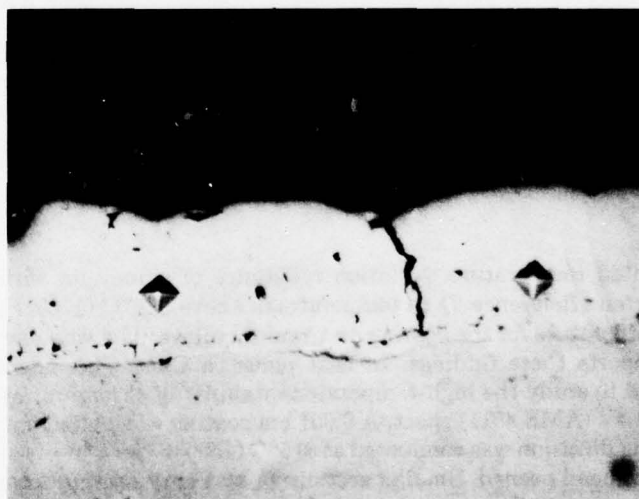
The penetration depth is 1/7th of the length of the impression diagonal. The impression diagonals (d) may be measured with an accuracy of 0.002 mm by means of a stage-microscope. The mean-value of several impressions is determined and the Vickers hardness specified in units of kgf/mm². In consequence, the Vickers hardness number (VHN) of a material is the relationship between the load (F) used to make the indentation and the remaining permanent impression of the square diamond-pyramid of a specific shape. Thus, the following equation may be related:

$$\text{VHN} = \frac{\text{load (F)}}{\text{surface (d)}} = \frac{F \times 1.8544}{d^2}$$

indentation

Test Results

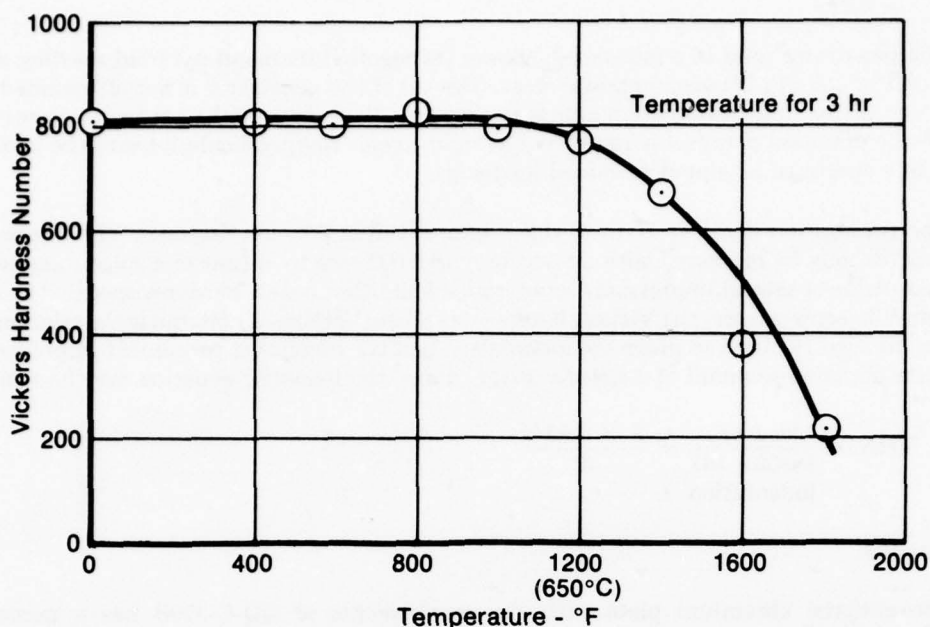
Conventional chromium plate with the requirements of QQ-C-320B has a minimum hardness of 600 VHN. Chromium-molybdenum deposits, applied as described for the 1% molybdenum in Appendix A, have a hardness of 780 VHN before heat treat (in the as-plated condition). After heat treat at 760°C (1400°F) for 3 hr, this hardness value drops to 650 VHN and subsequent heat treat below 760°C (1400°F) has no further effect on the hardness. Figure 11 shows typical diamond pyramid indentations.



FD 137309

Figure 11. Typical Diamond Pyramid Indentations

Figure 12 illustrates the effect of a 3-hr heat treat on coating hardness as a function of the heat-treat temperature. At higher temperatures, 980°C (1800°F), the hardness was reduced and dropped below 250 VHN.



FD 135818

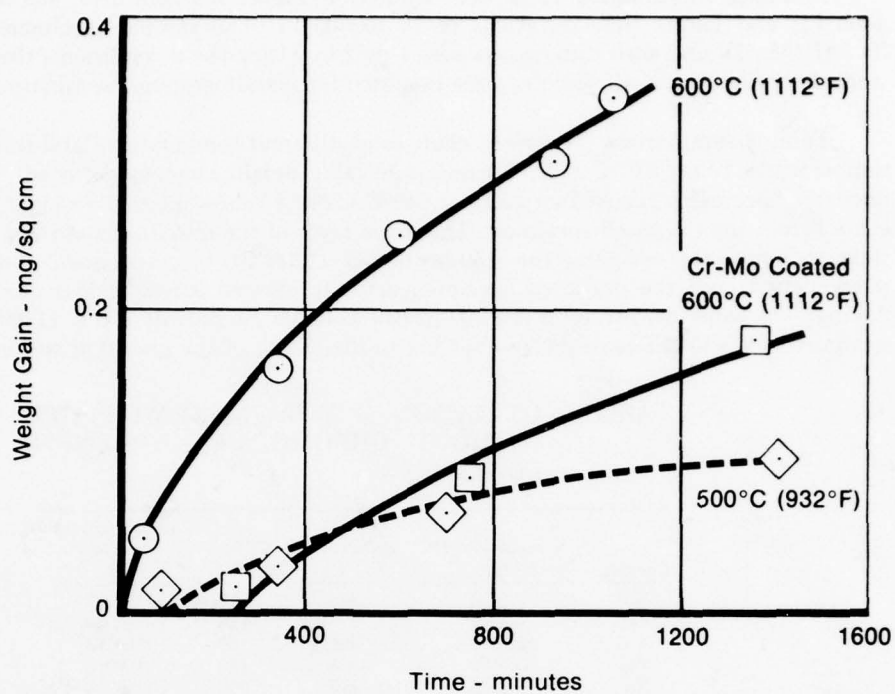
Figure 12. Relationship Between Heat-Treat Temperatures and Hardness

In a separate test, a pulse-plated chromium molybdenum coated specimen was prepared using an on/off cycle time of 3 ms. This current pulse-time was chosen because it provides the highest concentration of molybdenum in the deposit. The as-plated deposit had a hardness of 330 VHN, significantly lower than the direct-current plated alloys. The hardness of the Cr-Mo coating after heat treating at 1400°F for 3 hr was 291 VHN. The effect this reduced hardness has on the wear resistance is not known, however, the discovery should make possible the deposition of coatings with a gradient of hardness across its thickness by simply varying the current pulse duration during the plating operation. The advantages of such a gradient coating should include higher ductility, better adhesion and thermal stability.

OXIDATION

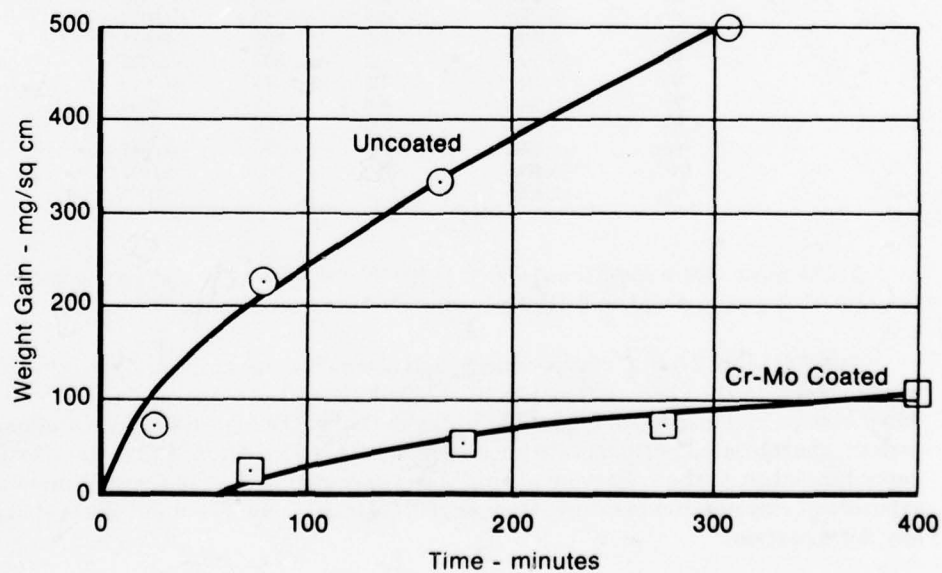
Excellent elevated temperature oxidation resistance of chromium with 5% molybdenum alloy has been reported (Reference 7) to temperatures above 927°C (1700°F), greater than the highest expected temperature for the coating on titanium alloys. The work completed in Phase 1 of this program supports these findings. In that phase, a Cahn Thermogravimetric Analysis (TGA) Unit was used to study the high-temperature stability of chromium-molybdenum coated and uncoated Ti 6Al-4V (AMS 4911) sheet. A 0.001 cm coating was plated onto a 6 by 10 by 0.03 cm specimen. Coating diffusion was conducted at 815°C (1500°F) for 1 hr in vacuum and then the coating was 5N2 glass bead peened. Smaller sections (1 by 1 cm) were cut from this panel so that the total weight did not exceed the 1 g electrobalance maximum capacity. For comparison, an uncoated sheet of AMS 4911 was also peened and cut into similar size sections. Weight gains were individually monitored using a recording electrobalance for approximately 24 hr. The results are shown in Figures 13 and 14.

In both cases the oxidation rates at 500°C (932°F) were low and did not seem to depend on whether the specimens were coated or uncoated. At 600°C, the differences between coated and uncoated specimens became more evident. The Cr-Mo coated pieces oxidized at approximately the same rate as they did at 500°F (1112°F), while uncoated titanium oxidized at a much greater rate. Raising the temperature to 700°C (1292°F) showed an even larger separation between coated and uncoated titanium. The coating improved the oxidation resistance at those temperatures.



FD 111390A

Figure 13. Oxidation Rate of Coated and Uncoated Ti 6Al-4V



FD 1113526A

Figure 14. Oxidation of Coated and Uncoated Ti 6Al-4V at 700°C (1292°F)

In Phase II, oxidation rates were studied at higher temperatures, 425 to 760°C (800 to 1400°F), and longer time durations of 15 to 300 hr. The sample specimens were made of Ti-8Al-1Mo-1V and their dimensions were 1 by 2 in. Using these conditions, the weight changes were determined, and all samples were mounted for metallographic examination.

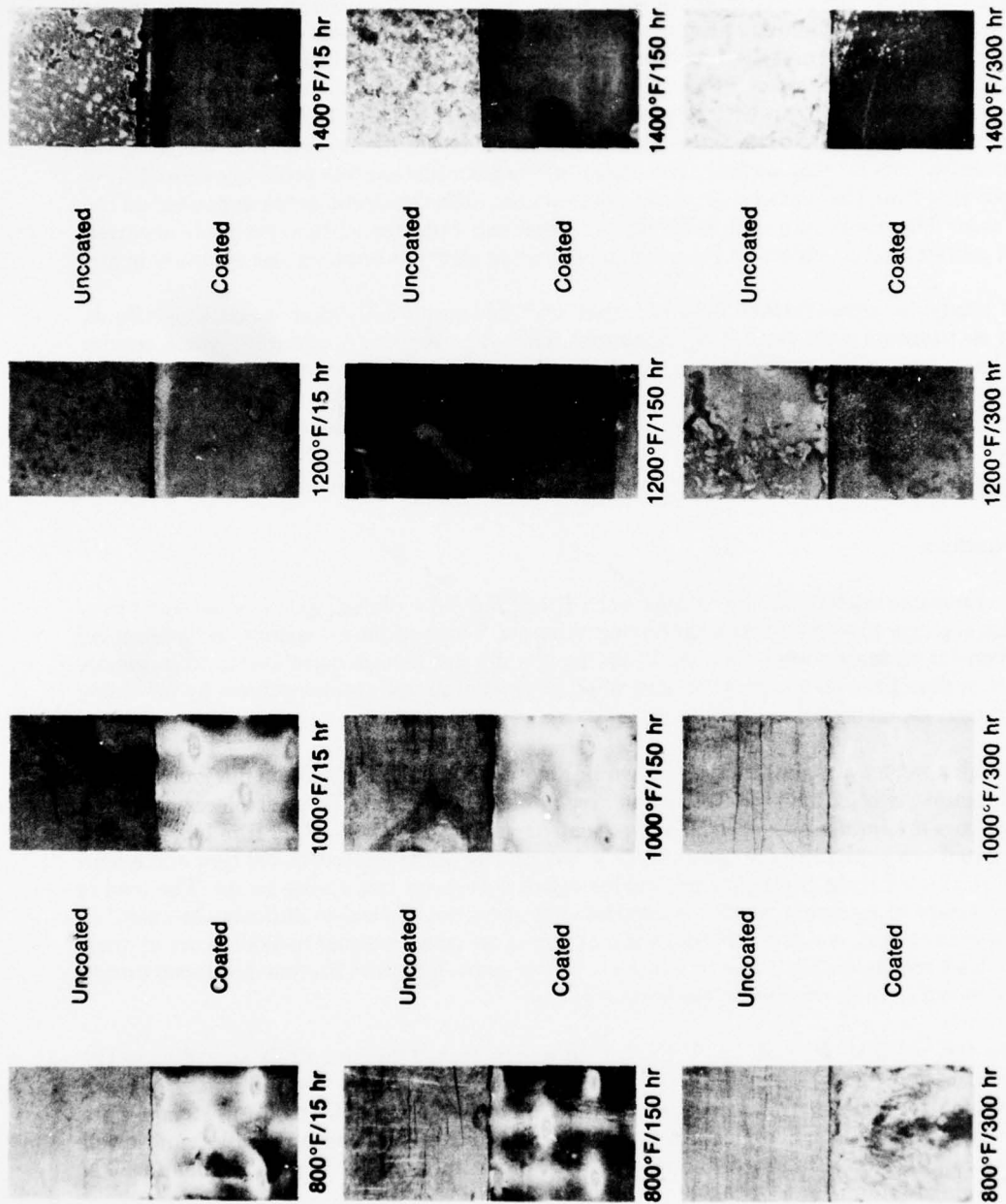
Table 2 summarizes the weight changes at different temperatures and time durations. At temperatures below 538°C (1000°F) no appreciable weight changes occurred. The coated and uncoated specimens exposed for 15 hr at 649°C (1200°F) show more of a weight increase for the coated than the uncoated specimen. The oxide layer of the uncoated specimen had scaled off, thus reducing the weight. The 150-hr 649°C (1200°F) test specimen showed the same phenomenon, and the uncoated specimen actually showed a weight loss due to scaling and flaking. The same held true for the 300-hr 649°C (1200°F) test. At 760°C (1400°F), the coated specimen also showed a weight loss but not to the extent of the uncoated specimen.

TABLE 2. OXIDATION OF Ti-8Al-1Mo-1V WITH AND WITHOUT CHROMIUM-MOLYBDENUM COATING

Coating	Sample Weight (g)	Temperature (°C)	Time (hr)	Weight Gain Or Loss (g)
No	5.2108	425	15	+0.0005
Yes	5.5443	425	15	+0.0004
No	5.2572	425	150	+0.0015
Yes	5.5829	425	150	+0.0012
No	5.1633	425	300	+0.0025
Yes	5.3047	425	300	+0.0023
No	5.3669	540	15	+0.0008
Yes	5.6721	540	15	+0.0007
No	5.1951	540	150	+0.0019
Yes	5.4975	540	150	+0.0025
No	5.1883	540	300	+0.0032
Yes	5.2561	540	300	+0.0036
No	5.1865	540	15	+0.0003
Yes	5.4629	650	15	+0.0062
No	5.2258	650	150	-0.0008
Yes	5.4062	650	150	+0.0016
No	5.0242	650	300	-0.0011
Yes	5.5468	650	300	+0.0022
No	5.2181	760	15	-0.0016
Yes	5.3996	760	15	-0.0009
No	5.0688	760	150	-0.0003
Yes	5.6693	760	150	-0.0047
No	5.4895	760	300	-0.1192
Yes	5.4895	760	300	-0.0050

At the most severe conditions, 760°C (1400°F) for 300 hr, the coated specimen had a weight loss of 0.0050 g as opposed to 0.1192 g for the uncoated specimen.

Another set of 2.5 by 5 cm specimens, half of which were coated and the other half uncoated, were exposed to the same parameters as the first set. After photographing the specimens were vapor blasted and inspected. Figure 15 illustrates that at the higher temperatures and longer time periods, the uncoated portion of the specimens show both oxidation and scaling. Inspection of the vapor blasted parts showed severe pitting of the uncoated portion of the specimens after the long exposure at elevated temperature. Only at 760°C (1400°F) for 300 hr did the coated portion show any deterioration.



FD 14354

Figure 15. Appearance of Coated and Uncoated Titanium Alloy After Elevated Temperature Exposure in Air

Metallographic examination of cross sections at up to 1000X showed no evidence of oxygen intrusion in either the coated or uncoated specimens at temperatures up to 538°C (1000°F). The uncoated specimen heated to 760°C (1400°F) for 150 hr exhibited greater α case formation (Figure 16) whereas the coated specimen did not.

To ascertain whether there was any alloy depletion of the titanium alloy, X-ray images were taken. Figure 17 shows the X-ray image of the Cr-Mo coating after heating at 760°C (1400°F) for 150 hr. Chromium, molybdenum, titanium, and calcium were all present on the surface of the sample. The calcium contamination probably came from the heat treat furnace. Figure 18 shows the X-ray image of the uncoated Ti-8Al-1V-1Mo substrate and surface. Comparing the surface layer of the uncoated specimen with the bulk material shows that significant depletion of both aluminum and molybdenum occurred as seen in Figure 19. In addition, chromium was detected in low concentrations on this surface. The chromium contamination was probably caused from metal vaporizing from the plated side, during the vacuum diffusion cycle, being deposited on the uncoated side. The aluminum and molybdenum depletion from the surface probably occurred because of preferential oxidation of those elements during elevated temperature exposure in air.

This study provides further evidence that the chromium-molybdenum coating affords protection to titanium with respect to oxidation. Also with respect to oxidation, these results indicate that a chromium-molybdenum coating will elevate titanium use temperatures by at least 100°C (212°F). This could prove to be of particular value on new titanium alloys exhibiting good high-temperature metallurgical stability but which do not possess sufficient oxidation resistant to allow extensive use.

WEAR TESTING

Test Procedure

The chromium-molybdenum coatings, both direct and pulse plated, were evaluated for wear and frictional properties on a Falex wear testing machine. This equipment is universally accepted as a method for screening wear couples. While results are not always correlatable to in-service conditions, it does provide a somewhat controlled method of materials comparison by providing fixed specimen geometry.

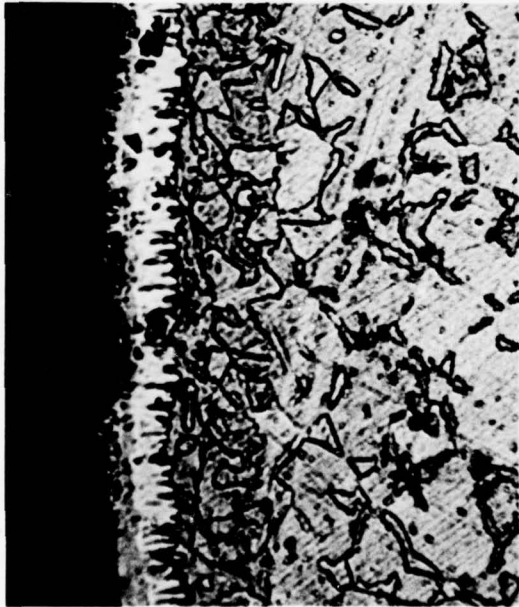
The Falex wear testing machine is shown in Figures 20 and 21. A 0.635-cm dia journal (pin) is rotated against two stationary V-blocks to give a four-line contact. The test pieces and their supporting jaws are immersed in the oil sample cup or run dry. Coatings or dry film lubricants to be investigated are applied to the pins and/or V-blocks. The pin is rotated at 290 rpm and a load is applied to the V-blocks through a nutcracker action lever arm and spring gauge. The load is applied by means of a ratchet wheel mechanism that also may be used to indicate the extent of wear like a micrometer. An 18-tooth advance on the ratchet wheel is equal to 0.00254 cm of wear. The entire load arm assembly is free to rotate about the main shaft, and friction developed during the test is shown in inch-pounds on the torque gauge.

In the test method selected, the V-block load was increased incrementally according to the schedule given in ASTM-D-2625 Procedure A. It consists of running two stationary V-block specimens against a rotating pin until a sharp increase of 1.13 Nm (10 in. to lb) in steady-state torque or pin breakage is experienced. A prescribed schedule of jaw load application and dwell time on the rotating pin is followed: 3 min at 1300 N plus 1 min at 2220 N plus 1 min at 3330 N plus 4450 N until failure.

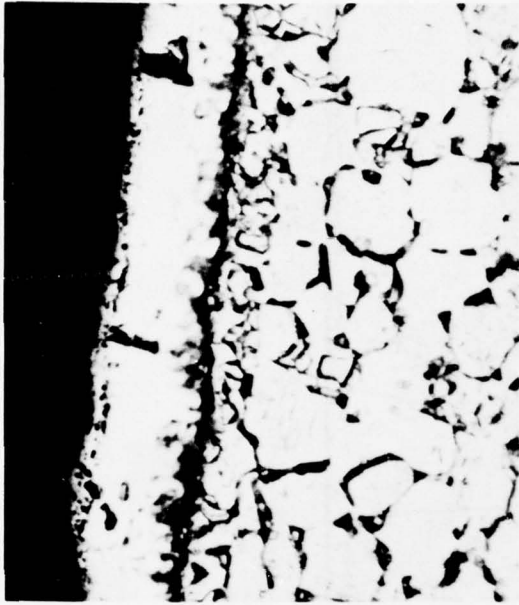
The pins and V-blocks used in this evaluation were Ti-6Al-4V or AISI 1137 steel. MIL-L-23699 aircraft turbine engine lubricant was used where lubricant was specified.

The test results are compiled in Table 3. In most instances, the titanium and steel alloy blocks and/or pins were coated with either the direct or pulse plating methods.

Uncoated



Coated



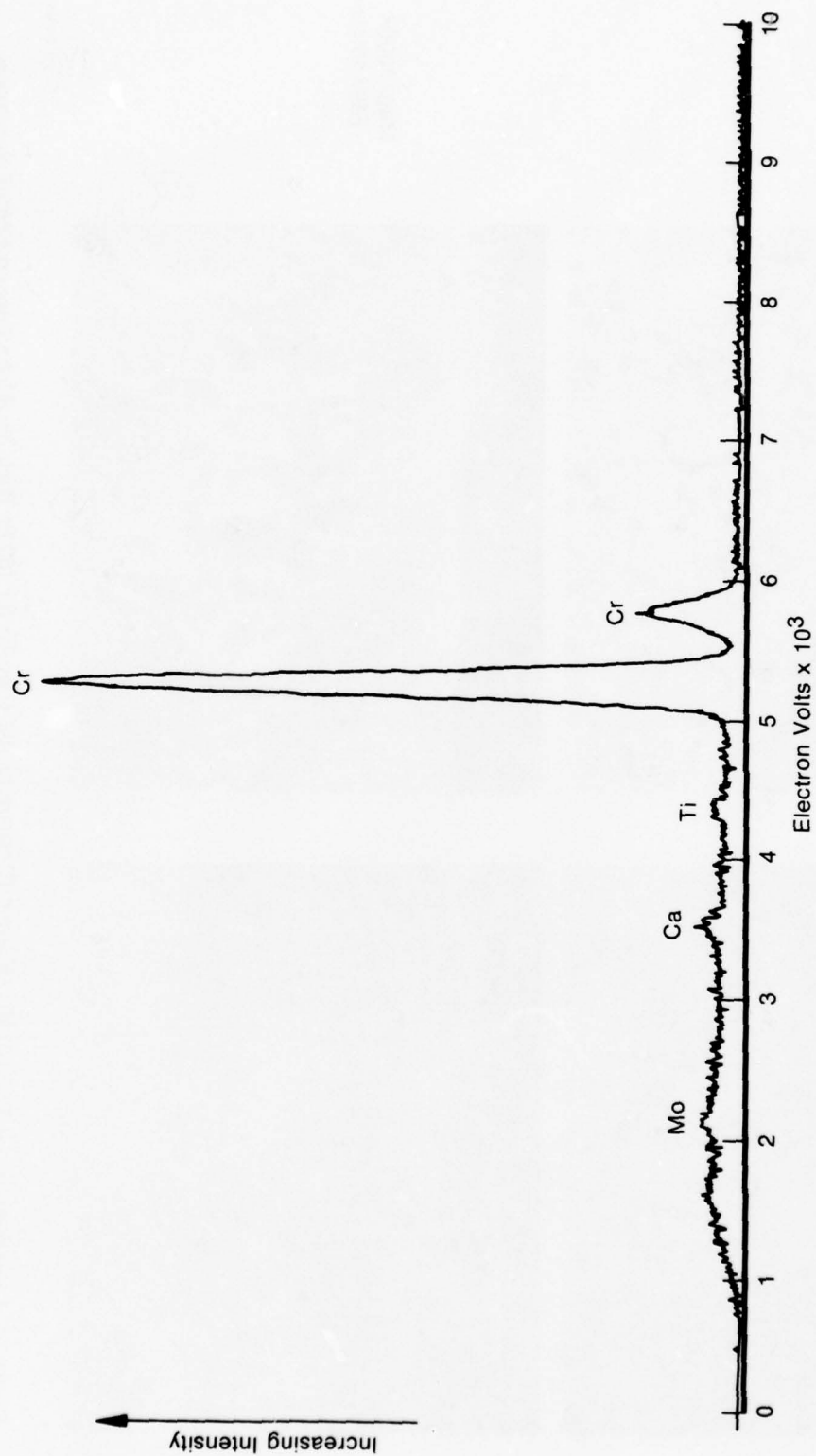
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Light Photo

Mag: 1000X
SEM Photo



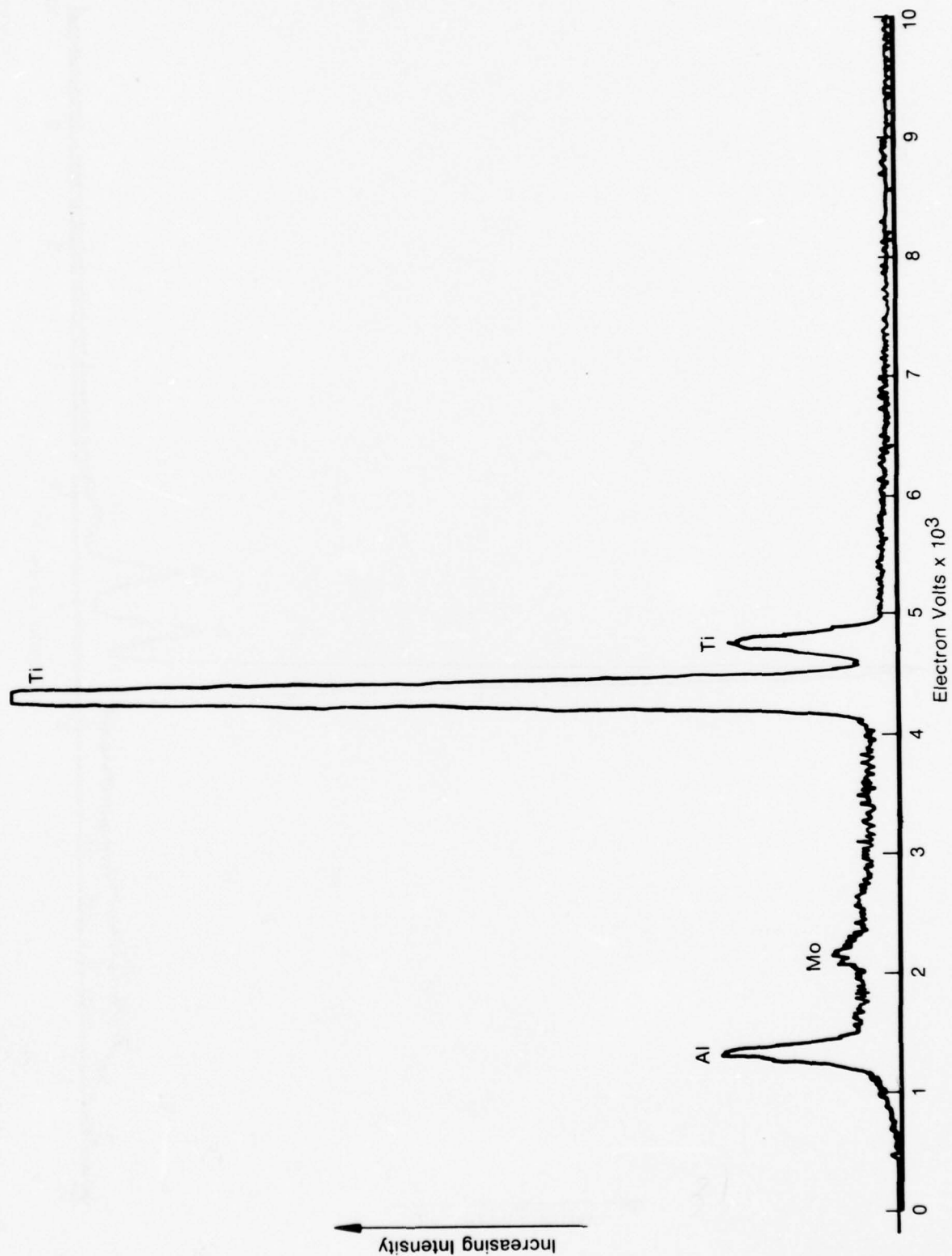
FD 137310

Figure 16. Cross Sections of Ti 8Al-1Mo-1V After Exposure to 760°C in Air for 150 hr With No Alpha Case on Coated Specimens



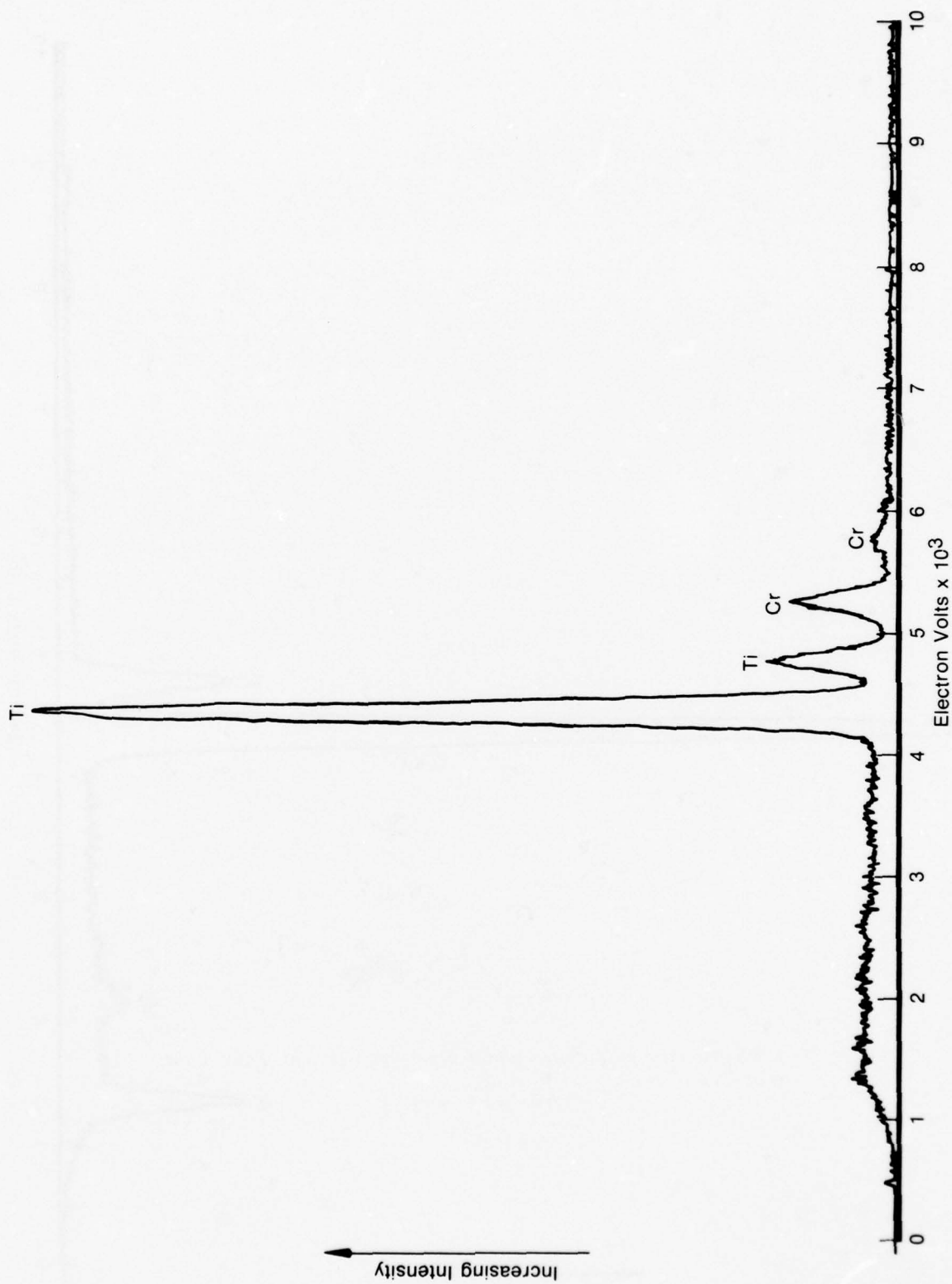
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Figure 17. X-Ray Image of Cr-Mo Coating After Baking at 760°C (1400°F) for 150 hr



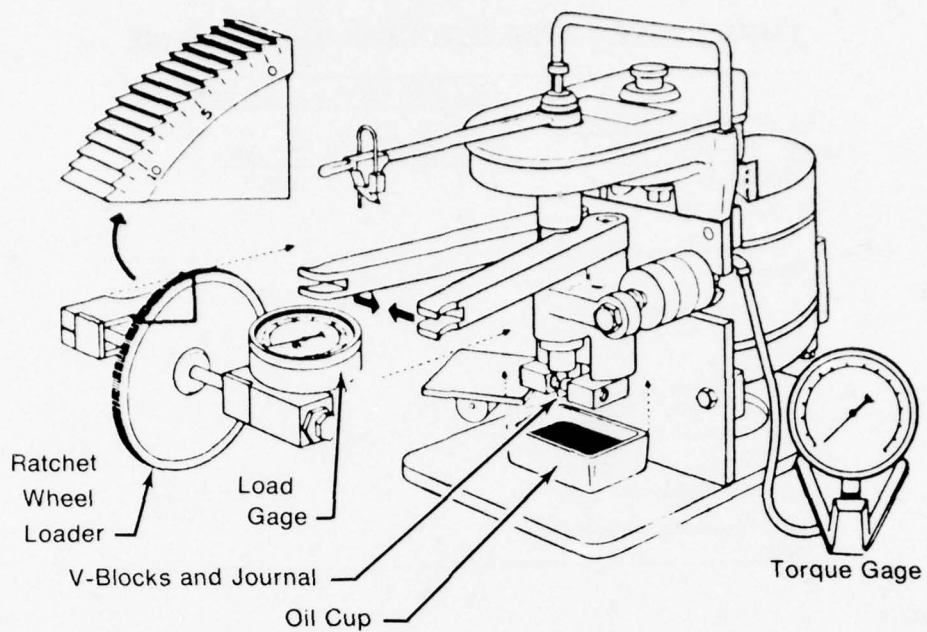
FD 147312

Figure 18. X-Ray Image of Titanium Substrate After Baking at 760°C (1400°F) for 150 hr



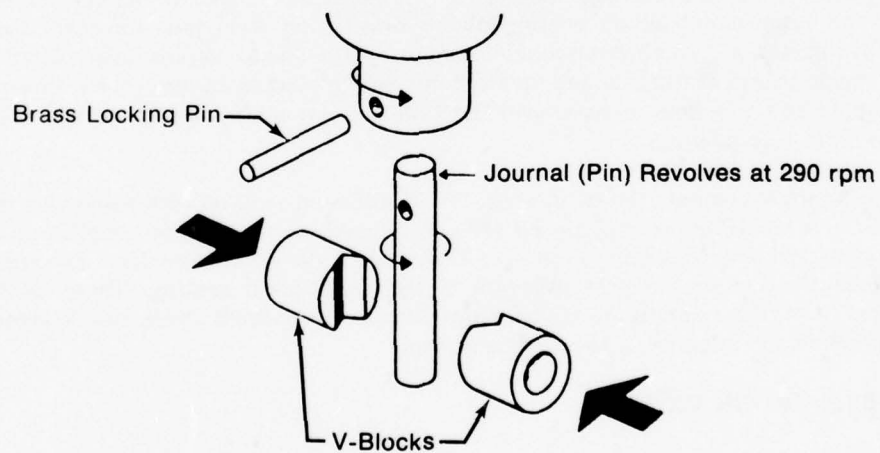
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Figure 19. X-Ray Image of Uncoated Titanium Surface After Baking at 760°C (1400°F) for 150 hr



FD 113527A

Figure 20. Schematic Diagram of Falex Lubricant Tester



FD 113528

Figure 21. Exploded View of V-Blocks and Journal Arrangement, Falex Lubricant Tester

TABLE 3. FALEX WEAR TEST USING MIL-L-23699 OIL

Test Material Combination	Minutes To Failure At Specified Loads			
	1330 N (300 lbf)	2200 N (500 lbf)	3330 N (750 lbf)	4450 DN (1000 lbf)
<i>Steel V-Block/Steel Pin</i>				
1. Bare Steel	3	1	Failed	
2. Chromium Plate	3	1	1	29
3. 1% chromium-molybdenum Direct	3	1	1	86
4. 1% chromium-molybdenum Pulsed at 3 ms ON and 3 ms OFF	3	1	1	169
<i>Steel V-Blocks/Titanium Pins</i>				
5. Bare Titanium	3	Failed		
6. Pulse Plated 1% chromium- molybdenum (no heat treat)	3	1	1	0.9
7. Pulse Plated/760°C 3 hr heat treat	3	1	1	118

Test Results

Steel against steel failed the wear test occasionally before completing the 1 min at 3300 N (750 lbf). However, the chromium-molybdenum coatings substantially improved the wear life. These coatings were produced using the electrolyte described in Appendix 3. All test specimens were run to failure to ascertain the beneficial effects of the chromium-molybdenum coating. The individual results in Table 3 represent the average of at least three tests. As can be seen from the table, the chromium-molybdenum coating plated directly on steel pins improves the wear resistance by a factor of 3 over conventional chromium plate. The steel pins pulse-plated with a chromium-molybdenum coating showed a sixfold increase in wear resistance over a conventional chromium plate and a twofold increase over the wear protection afforded by the direct-current plated chromium-molybdenum.

In an attempt to evaluate the effect of the 760°C diffusion cycle on wear resistance, a series of wear tests were conducted using diffused and nondiffused specimens. The nondiffused coated titanium pins lasted only 53 sec as compared to 118 min for the diffused coating. The reason for this is apparently due to the poor adhesion of the nondiffused coating. These wear tests demonstrated that the chromium-molybdenum diffusion heat-treat cycle has a significant beneficial effect on the integrity of the coating system.

COATING EVALUATION ON STEEL

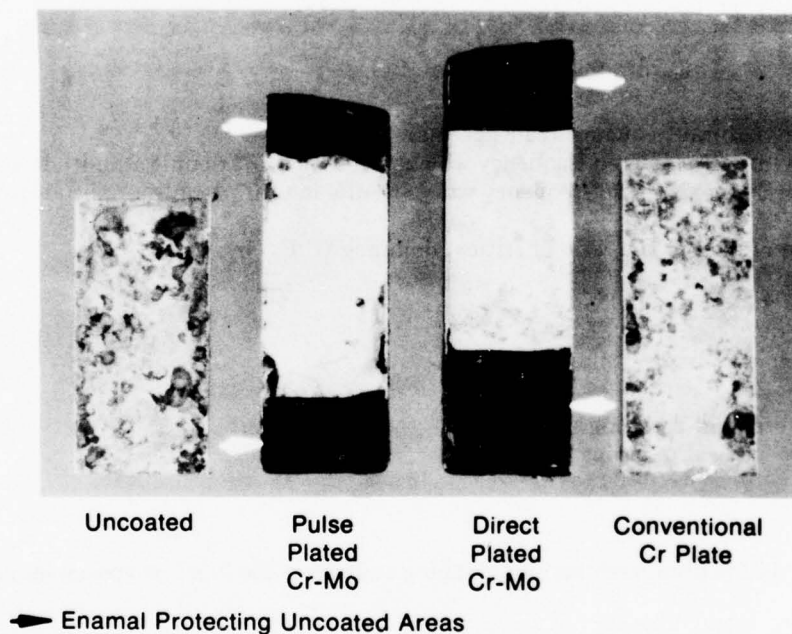
Chromium has long been used as the conventional wear-resistant coating for steel. However, because of the formation of surface cracks, inherent in the plating process, corrosion resistance of the resultant coatings is very poor. Traditionally, to achieve corrosion resistance, undercoatings of nickel or copper-nickel are applied prior to the chromium plate. This nickel underplate, however, cannot be used in critical bearing steel applications because it is softer than steel and chromium and thus results in bearing deformation under high load. By omitting the nickel underlayer, the bearing corrosion resistance is significantly reduced.

The chromium-molybdenum coating on steel has shown better wear resistance than chromium plating. Since this coating is also crack-free, it could potentially provide better corrosion protection to steel than conventional chromium plating. To verify this protection, a salt-spray corrosion test was performed per ASTM B-117.

Four specimens of a typical bearing alloy, AMS 6431B, were prepared for this study as:

- Specimen No. 1 — Uncoated
- Specimen No. 2 — Pulse-plated chromium-molybdenum, 3% Mo
- Specimen No. 3 — Direct-current chromium-molybdenum plated, 1% Mo
- Specimen No. 4 — Chromium plate, 0.5 mils thickness

These specimens were placed in a salt-spray cabinet for 8 hr as shown in Figure 22. Results of this study indicate a direct-current chromium-molybdenum coating will provide greater corrosion protection than that afforded by a conventional chromium plate. Additionally, the pulse-plated chromium-molybdenum specimen demonstrated more protection than either the direct-current chromium-molybdenum coating or the conventional chromium plate. These results indicate that for many applications a pulse-plated chromium-molybdenum coating can replace a conventional chromium plate and provide increased corrosion protection.



FE 164950A

Figure 22. Appearance of Coated and Uncoated AMS 6431 After Salt Spray Test

MODULUS AND DAMPING CHARACTERISTICS

This part of the test program was conducted to evaluate the effect of the chromium-molybdenum coating on the modulus and damping characteristics of Ti 8Al-1Mo-1V base alloy. Such information is important for those applications in which the natural frequency and vibrational characteristics can influence the part-life, for example, of a gas turbine compressor blade.

Test Procedure

Four rectangular cross section bars 0.292 by 2.54 by 20 cm (0.115 by 1.0 by 8.0 in.) were fabricated from Ti 8Al-1Mo-1V single-annealed material. All four bars were tested for modulus

and damping characteristics in the as-received condition. Two specimens were then vapor blasted, etched and plated with chromium-molybdenum coating. All four bars (two plated, two not plated) were heat treated at 760°C (1400°F) for 3 hr in vacuum followed by a light vapor blast. The four bars were then retested to determine the effects of the coating and to allow separation of heat-treat effects from coating effects. All testing was performed on specimens in the cantilever first bending mode.

Static modulus determination was accomplished using the relation:

$$\text{Young's Modulus (E}_{\text{static}}) = \frac{\text{Strain (experimental)}}{\text{Stress (MC/I calculated)}}$$

Dynamic moduli were calculated using a relationship based on specimen natural frequency, density, and geometric parameters.

Damping determination (Q) was accomplished using the relation:

$$Q = fn/f_2 - f_1$$

where,

- fn = natural frequency (cantilever first bending)
- f₂ = upper sideband frequency which results in 0.707 double amplitude at fn.
- f₁ = lower sideband frequency which results in 0.707 double amplitude at fn.

Q can be related to the ratio of critical damping (C/C_c) by:

$$1/Q = 2 C/C_c$$

where,

- C = actual damping of specimen
- C_c = critical damping of specimen

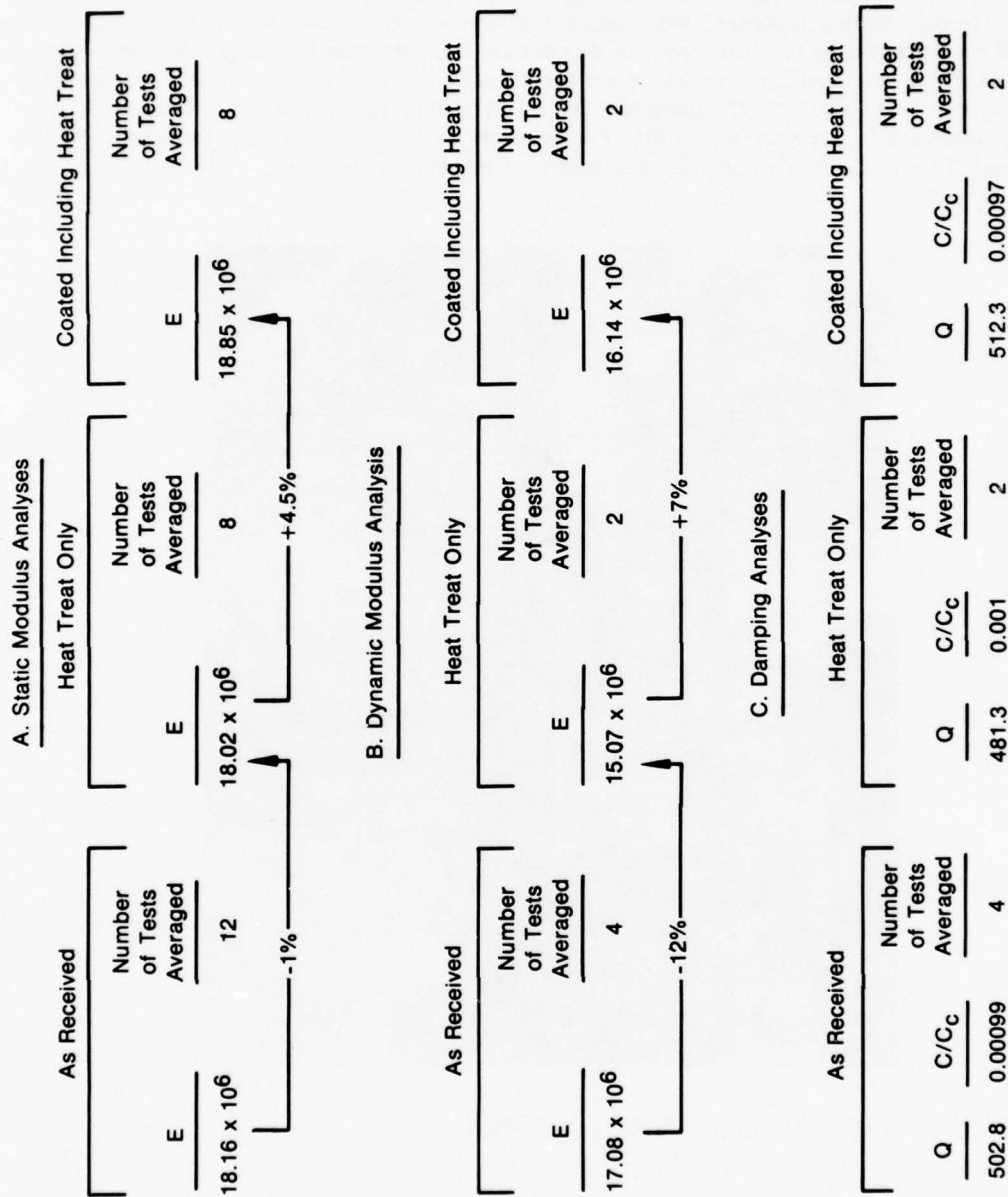
Test Results

Average results from modulus and damping analysis of the four test specimens are shown in Figure 23.

After heat treating at 760°C (1400°F) for 3 hr, static and dynamic modulus decreased by an average of 1 and 12%, respectively. These decreases may be attributed to either the annealing-type cycle of the heat treat or the slight metal removal and peening effect of the vapor blast operation used to remove surface oxidation after heat treat.

Comparing the heat treat only results with the chromium-molybdenum coated results show average increases in static and dynamic moduli of 4.5 and 7%, respectively. Apparently, the coating does have a slight stiffening effect on structures although this effect is so small it is probably of no practical significance.

The heat treat and the coating process had little effect on specimen damping (Figure 23c). Heat treating produced a slight damping increase with a subsequent decrease after plating; however, these effects were essentially insignificant.



FD 137317

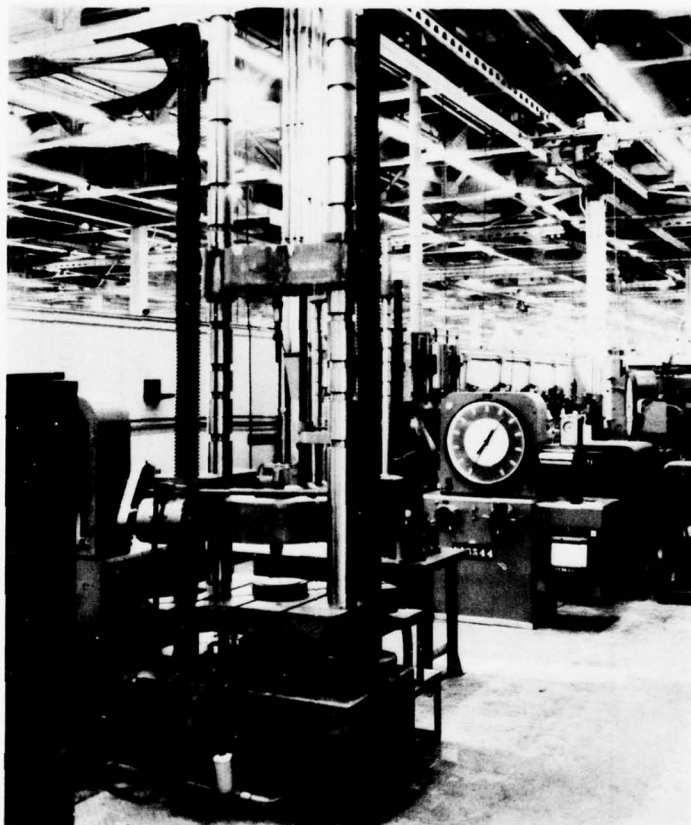
Figure 23. Test Specimens With 0.0015 in. Thick Chrome-Molybdenum Coating

COATING EFFECT ON MECHANICAL PROPERTIES

Tensile Strength, Yield Strength and Elongation

Test Procedure

The most popular method of determining mechanical properties is the tensile test. The two types of tensile testing machines are: mechanical screw and hydraulic. Figure 24 shows the hydraulic tensile testing machine used. In this device, the specimen is clamped between a fixed and a movable crosshead. A tensile or stretching stress is applied by causing the movable crosshead to move away from the fixed one. In this hydraulic machine, a motor causes a fluid to develop hydrostatic pressure on a piston which acts as the force causing movement of the crosshead. This pressure also provides the means of measuring the load.



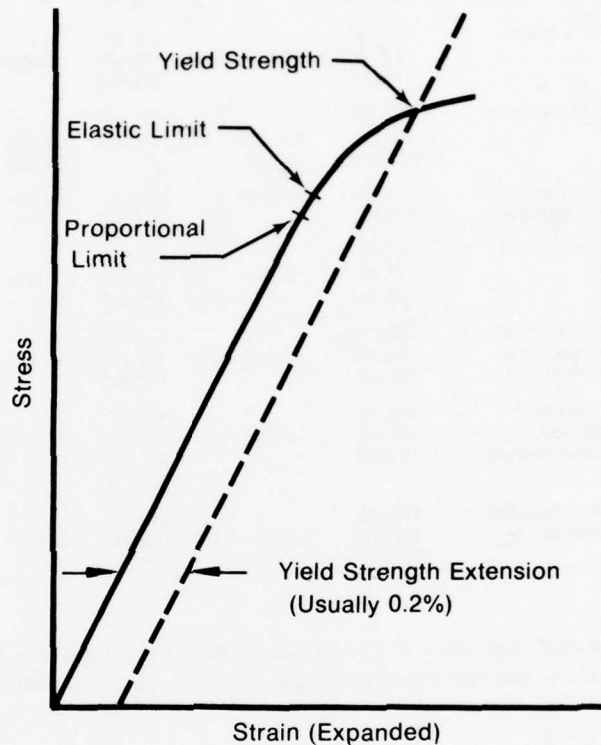
FC 8321

Figure 24. Tensile Testing Equipment

Devices to measure strain (the elongation) are independent of the tensile testing machine equipment. For very small strains (elastic deformation), two methods are in common use. One is a special mechanical-electrical instrument called a high magnification extensometer which is clamped to the specimen. The other is an electrical resistance wire strain gage which is cemented to the specimen and senses the small deformations of the specimen by changes in its electrical

resistance. When used with a sensitive electronic amplifier and bridge network, an accurate measure of strain is obtained. The electronic strain gage is more sensitive to small elastic strains than the extensometer and can be readily attached to odd-shaped test specimens.

Of greater importance is the yield strength which is defined as the stress at which some predetermined amount of permanent strain occurs, usually 0.2%. Figure 25 illustrates this technique in which the yield strength is found by drawing a parallel line at 0.2% strain.



FD 137314

Figure 25. Technique for Determining Yield Strength

Test Results

Table 4 summarizes results of tensile testing of 26 AMS 4911 (Ti-6Al-4V) specimens which had been subjected to various processing treatments. These are compared with as-machined (no plate/no heat-treat) control specimens. Three heat-treat atmospheres, air, argon and vacuum are compared as to their effect on tensile properties (items 2, 3, and 4). The effect of wet abrasive blasting subsequent to air heat treating is shown in item 6. Plating/stripping/replating/heat treating in air or argon effects are shown in items 5 and 9. The importance of items 5 and 9 show that expensive engine parts can be stripped and replated if the desired results are not achieved.

The influence of duplex plating (two coats of plate with or without intermediate heat treatment) is listed in items 7 and 8.

No significant reduction in tensile strength or elongation was observed. The most severe effect was noted on yield strength of air heat-treated, uncoated specimens.

TABLE 4. TENSILE AMS 4911

Item	Offset	\bar{X}	2σ	Tensile	\bar{X}	2σ	El (%)	\bar{X}	2σ
	YS(0.2% psi)			Strength psi					
1. No plate or heat treat.	131,000			136,800			11.0		
	131,000	130,767	472	136,700	136,300	781	10.0	10.3	0.58
	130,300			135,400			10.0		
2. Heat treated in air.	120,700			132,000			10.0		
	124,100	122,600	1735	133,500	133,267	1168	10.0	10.3	0.58
	123,000			134,300			11.0		
3. Heat treated in Argon.	126,300			136,400			10.0		
	128,100	128,133	1850	136,000	135,800	721	10.0	9.8	0.29
	130,000			135,000			9.5		
4. Heat treated in vacuum.	129,400			135,500			9.5		
	129,600	128,933	987	136,000	135,300	818	9.3	9.8	0.64
	127,800	27,800			134,400			10.5	
5. Plated, stripped, replated and heat treated in air.	124,100	124,850		132,000	132,000	0	12.0	11.8	
	125,600			132,000			11.5		
6. Heat treated in air and vapor blasted.	127,200			133,700			9.3		
	127,200	127,200	0	134,000	133,666	351	11.0	10.1	0.85
	127,200			133,300			10.0		
7. Duplex coat with no heat treat after second coat (heat treated in Argon).	126,100			134,700			9.3		
	127,200	126,366	737	134,400	133,933	1079	11.3	10.3	1.0
	125,800			132,700			10.3		
8. Duplex coat with heat treat after first and second coat (heat treated in Argon).	126,500			134,300			10.5		
	125,300	125,800	625	133,600	133,867	379	11.0	10.8	0.25
	125,600			133,700			10.8		
9. Plated, stripped, replated and heat treated in Argon.	126,500			136,300			12.0		
	127,600	126,967	569	138,000	136,967	907	10.0	10.8	1.04
	126,800			136,600			10.5		

It is concluded that, with the exception of heat treating in air, the tensile strength of AMS 4911 titanium alloy remains unaffected by processing steps required for chromium-molybdenum coating.

High-Cycle Fatigue

Test Procedure

For most coated titanium alloy applications, it is important that the coating does not compromise fatigue strength. Previous, preliminary testing of a small number of rotating beam specimens of Ti 8Al-1Mo-1V (PWA 1202) at 482°C (900°F) had shown that chromium-molybdenum does not reduce fatigue strength. In the first phase (Reference 1), fatigue specimens were machined from Ti 8Al-1Mo-1V (AMS 4916) sheet stock. These flat, sheet fatigue specimens also showed no reduction in fatigue strength when coated with chromium-molybdenum.

During this phase, a test program was undertaken to more completely characterize the coating effect on fatigue strength. Table 5 shows how the specimens were prepared to support this study. (See Figures 26 and 27.)

After machining, all specimens were stress relieved at 590°C (1100°F) for 2 hr. Coatings were subsequently applied per Appendix A.

TABLE 5. FATIGUE SPECIMEN PREPARATION

Number of Specimens	Specimen				
	Material		Configuration		
24	Ti-6Al-4V	(AMS 4928)	Notched	($K_t=2$)	(Figure 27)
24	Ti-8Al-1V-1Mo	(PWA 1202)	Notched	($K_t=2$)	(Figure 27)
24	Ti-6Al-4V	(AMS 4928)	Smooth	($K_t=1$)	(Figure 26)
36	Ti-8Al-1V-1Mo	(PWA 1202)	Smooth	($K_t=1$)	(Figure 26)

Test Results

Specimens were tested in bending on Krouse rotating beam fatigue machines at approximately 3600 rpm to failure (or 10^7 cycles) at various test temperatures. All failed specimens had the failure originating in the gage area of the specimen.

Tables 6 and 7 and Figures 26 and 27 summarize the results of this test series. The Ti 8Al-1Mo-1V (PWA 1202) fatigue strength (Figure 26) was reduced after processing per the coating procedure but the reduction turned out to be caused by the heat treat cycle used for diffusion.

Figure 28 illustrates the results of testing the Ti 6-4 (AMS 4928). The uncoated specimens had strength levels near the minimum of previously run tests while the coated were slightly below this minimum. The notched ($K_t = 2$) tests showed essentially no difference in coated and uncoated fatigue strength levels.

Discussion of Results

Tests run during the first program phase (Reference 1) used annealed Ti-8Al-1Mo-1V (AMS 4916). The chromium-molybdenum diffusion cycle, equivalent to an additional annealing cycle, did not adversely affect the alloy fatigue strength.

The Ti-8Al-1Mo-1V fatigue specimen alloy used in this phase was originally in the fully heat-treated condition (solution at 900 to 1000°C for 1 hr followed by a stabilizing anneal at 550 to 600°C for 8 hr). The additional annealing cycle imposed on the fully heat-treated material, as a result of the chromium-molybdenum diffusion operation, degraded fatigue properties. (See Figure 29.) Even though the coated results were still above the minimum curve previously obtained from testing several heats of fully heat-treated Ti-8Al-1Mo-1V, the degradation should be avoided if possible. Early tests conducted on coated, fully heat-treated Ti-8Al-1Mo-1V using a diffusion cycle of 980°C for 1 hr followed by an 8 hr aging cycle at 575°C showed the alloy could be coated without affecting the fatigue strength.

To minimize the effects of the coating process on the fatigue strength of titanium alloys, it is necessary to select a diffusion heat-treat cycle compatible with previous heat treatment given the material. In addition, to minimize distortion, while providing adequate adhesion, the temperature-time combination must be selected to provide at least the diffusion depth obtained by the normal 760°C (1400°F) for 3 hr heat-treat. Further, it is desirable to minimize the total diffusion depth to as little as possible above that required for adhesion. This results in minimizing distortion effects occurring during the diffusion cycle.

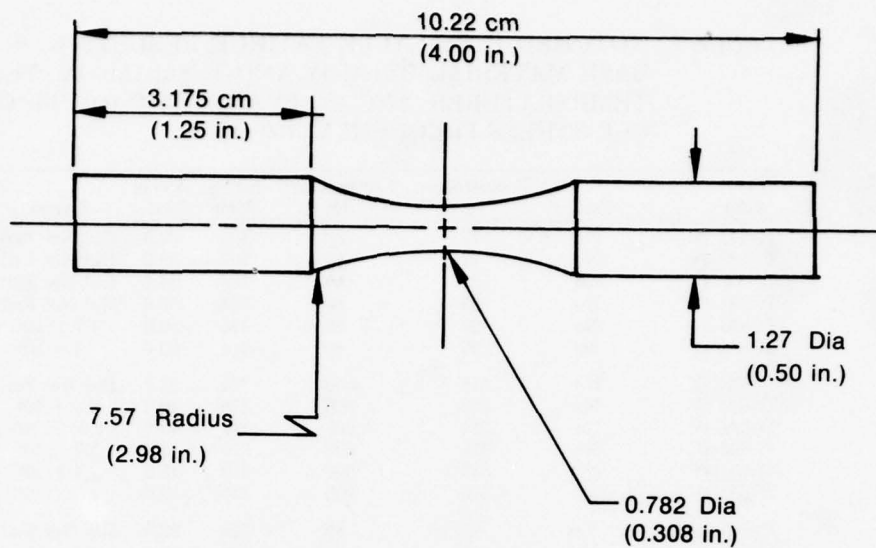
TABLE 6. HIGH-CYCLE FATIGUE RESULTS $K_T = 1$, BASE MATERIAL:
Ti-6Al-4V AND Ti-8Al-1Mo-1V TEST TEMPERATURES: 27°C
(80°F), 315°C (600°F), 482°C (900°F) AND 593°C (1100°F);
CYCLE STRESS FREQUENCY: 100 Hz

Alloy	Coated	Temperature °C	Temperature °F	Stress MPa	Stress ksi	Cycles
Ti-6Al-4V	No	27	80	345	50	Did Not Fail
Ti-6Al-4V	No	27	80	362	52.5	Did Not Fail
Ti-6Al-4V	No	27	80	379	55	2.5×10^6
Ti-6Al-4V	No	27	80	414	60	1.9×10^6
Ti-6Al-4V	No	27	80	483	70	1.55×10^6
Ti-6Al-4V	No	27	80	552	80	1.38×10^6
Ti-6Al-4V	No	315	600	345	50	Did Not Fail
Ti-6Al-4V	No	315	600	379	55	Did Not Fail
Ti-6Al-4V	No	315	600	414	60	1.0×10^6
Ti-6Al-4V	No	315	600	414	60	1.44×10^6
Ti-6Al-4V	No	315	600	483	70	1.29×10^6
Ti-6Al-4V	No	315	600	552	80	1.03×10^6
Ti-6Al-4V	Yes	27	80	276	40	Did Not Fail
Ti-6Al-4V	Yes	27	80	310	45	1.07×10^6
Ti-6Al-4V	Yes	27	80	379	55	1.05×10^6
Ti-6Al-4V	Yes	27	80	414	60	1.85×10^6
Ti-6Al-4V	Yes	27	80	517	75	1.3×10^6
Ti-6Al-4V	Yes	315	600	207	30	Did Not Fail
Ti-6Al-4V	Yes	315	600	276	40	1.04×10^6
Ti-6Al-4V	Yes	315	600	345	50	1.48×10^6
Ti-6Al-4V	Yes	315	600	379	55	1.43×10^6
Ti-6Al-4V	Yes	315	600	414	60	1.3×10^6
Ti-6Al-4V	Yes	315	600	483	70	1.04×10^6
Ti-8Al-1Mo-1V	No	27	80	414	60	Did Not Fail
Ti-8Al-1Mo-1V	No	27	80	414	60	Did Not Fail
Ti-8Al-1Mo-1V	No	27	80	427	62	1.9×10^6
Ti-8Al-1Mo-1V	No	27	80	448	65	1.65×10^6
Ti-8Al-1Mo-1V	No	27	80	462	67	1.85×10^6
Ti-8Al-1Mo-1V	No	27	80	483	70	2.5×10^6
Ti-8Al-1Mo-1V	No	482	900	324	47	Did Not Fail
Ti-8Al-1Mo-1V	No	482	900	345	50	4.5×10^6
Ti-8Al-1Mo-1V	No	482	900	414	60	Did Not Fail
Ti-8Al-1Mo-1V	No	482	900	414	60	2.3×10^6
Ti-8Al-1Mo-1V	No	482	900	427	62	3.5×10^6
Ti-8Al-1Mo-1V	No	482	900	448	65	4.0×10^6
Ti-8Al-1Mo-1V	No	482	900	448	65	7.2×10^6
Ti-8Al-1Mo-1V	No	593	1100	310	45	Did Not Fail
Ti-8Al-1Mo-1V	No	593	1100	345	50	Did Not Fail
Ti-8Al-1Mo-1V	No	593	1100	379	55	1.02×10^6
Ti-8Al-1Mo-1V	No	593	1100	448	65	0.45×10^6
Ti-8Al-1Mo-1V	Yes	27	80	324	47	1.05×10^6
Ti-8Al-1Mo-1V	Yes	27	80	345	50	1.6×10^6
Ti-8Al-1Mo-1V	Yes	27	80	379	55	Did Not Fail
Ti-8Al-1Mo-1V	Yes	27	80	379	55	3.75×10^6
Ti-8Al-1Mo-1V	Yes	27	80	414	60	1.55×10^6
Ti-8Al-1Mo-1V	Yes	27	80	448	65	1.65×10^6
Ti-8Al-1Mo-1V	Yes	482	900	324	47	Did Not Fail
Ti-8Al-1Mo-1V	Yes	482	900	379	55	8×10^6
Ti-8Al-1Mo-1V	Yes	482	900	379	55	4.5×10^6
Ti-8Al-1Mo-1V	Yes	482	900	483	70	2.3×10^6
Ti-8Al-1Mo-1V	Yes	593	1100	241	35	Did Not Fail
Ti-8Al-1Mo-1V	Yes	593	1100	276	40	Did Not Fail
Ti-8Al-1Mo-1V	Yes	593	1100	276	40	1.5×10^6
Ti-8Al-1Mo-1V	Yes	593	1100	310	45	5.5×10^6
Ti-8Al-1Mo-1V	Yes	593	1100	345	50	3.2×10^6
Ti-8Al-1Mo-1V	Yes	593	1100	379	55	4.5×10^6
Ti-8Al-1Mo-1V	Yes	593	1100	414	60	0.9×10^6
Ti-8Al-1Mo-1V	No*	482	900	379	55	3.1×10^6

*Uncoated with a 3 hour 760°C heat treat

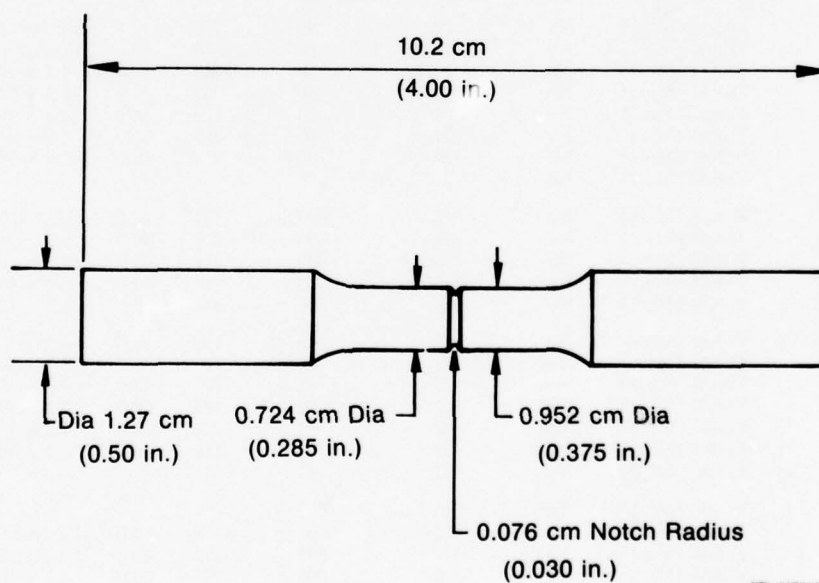
TABLE 7. NOTCHED HIGH-CYCLE FATIGUE RESULTS $K_T = 2$,
BASE MATERIAL: Ti-6Al-4V AND Ti-8Al-1Mo-1V, TEST
TEMPERATURES: 27°C (80°F) AND 315°C (600°F); CY-
CLE STRESS FREQUENCY: 100 Hz

Alloy	Coated	Temperature °C	Temperature °F	Stress MPa	Stress ksi	Cycles
Ti-6Al-4V	No	27	80	155	22.5	Did Not Fail
Ti-6Al-4V	No	27	80	207	30.0	Did Not Fail
Ti-6Al-4V	No	27	80	221	32.0	Did Not Fail
Ti-6Al-4V	No	27	80	224	32.5	Did Not Fail
Ti-6Al-4V	No	27	80	345	50.0	1.7×10^6
Ti-6Al-4V	No	27	80	414	60.0	1.4×10^6
Ti-6Al-4V	No	315	600	224	32.5	Did Not Fail
Ti-6Al-4V	No	315	600	276	40.0	4.4×10^6
Ti-6Al-4V	No	315	600	276	40.0	2.55×10^6
Ti-6Al-4V	No	315	600	310	45.0	3.7×10^6
Ti-6Al-4V	No	315	600	310	45.0	2.4×10^6
Ti-6Al-4V	No	315	600	345	50.0	1.1×10^6
Ti-6Al-4V	Yes	27	80	224	32.5	Did Not Fail
Ti-6Al-4V	Yes	27	80	228	33.0	1.5×10^6
Ti-6Al-4V	Yes	27	80	276	40.0	1.4×10^6
Ti-6Al-4V	Yes	27	80	276	40.0	7.6×10^6
Ti-6Al-4V	Yes	27	80	345	50.0	3.3×10^6
Ti-6Al-4V	Yes	27	80	421	61.0	1.3×10^6
Ti-6Al-4V	Yes	315	600	138	20.0	Did Not Fail
Ti-6Al-4V	Yes	315	600	172	25.0	1.5×10^6
Ti-6Al-4V	Yes	315	600	207	30.0	5.6×10^6
Ti-6Al-4V	Yes	315	600	276	40.0	1.9×10^6
Ti-6Al-4V	Yes	315	600	345	50.0	1.2×10^6
Ti-8Al-1Mo-1V	No	27	80	276	40.0	Did Not Fail
Ti-8Al-1Mo-1V	No	27	80	345	50.0	Did Not Fail
Ti-8Al-1Mo-1V	No	27	80	345	50.0	5.2×10^6
Ti-8Al-1Mo-1V	No	27	80	352	51.0	4.9×10^6
Ti-8Al-1Mo-1V	No	27	80	379	55.0	4.2×10^6
Ti-8Al-1Mo-1V	No	27	80	379	55.0	4.1×10^6
Ti-8Al-1Mo-1V	No	27	80	414	60.0	1.9×10^6
Ti-8Al-1Mo-1V	No	27	80			
Ti-8Al-1Mo-1V	No	315	600	241	35.0	Did Not Fail
Ti-8Al-1Mo-1V	No	315	600	276	40.0	2.5×10^6
Ti-8Al-1Mo-1V	No	315	600	310	45.0	4.2×10^6
Ti-8Al-1Mo-1V	No	315	600	345	50.0	3.2×10^6
Ti-8Al-1Mo-1V	No	315	600	414	60.0	3.0×10^6
Ti-8Al-1Mo-1V	Yes	27	80	138	20.0	Did Not Fail
Ti-8Al-1Mo-1V	Yes	27	80	207	30.0	2.5×10^6
Ti-8Al-1Mo-1V	Yes	27	80	276	40.0	6.0×10^6
Ti-8Al-1Mo-1V	Yes	27	80	345	50.0	2.9×10^6
Ti-8Al-1Mo-1V	Yes	27	80	379	55.0	1.6×10^6
Ti-8Al-1Mo-1V	Yes	27	80	414	60.0	1.2×10^6
Ti-8Al-1Mo-1V	Yes	27	80			
Ti-8Al-1Mo-1V	Yes	315	600			
Ti-8Al-1Mo-1V	Yes	315	600	138	20.0	Did Not Fail
Ti-8Al-1Mo-1V	Yes	315	600	172	25.0	Did Not Fail
Ti-8Al-1Mo-1V	Yes	315	600	207	30.0	1.2×10^6
Ti-8Al-1Mo-1V	Yes	315	600	276	40.0	2.8×10^6
Ti-8Al-1Mo-1V	Yes	315	600	345	50.0	2.3×10^6
Ti-8Al-1Mo-1V	Yes	315	600	414	60.0	1.1×10^6



FD 137318

Figure 26. Smooth Krouse Rotating Beam Fatigue Specimen, $K_T = 1$



FD 137319

Figure 27. Notched Fatigue Specimen, $K_T = 2$

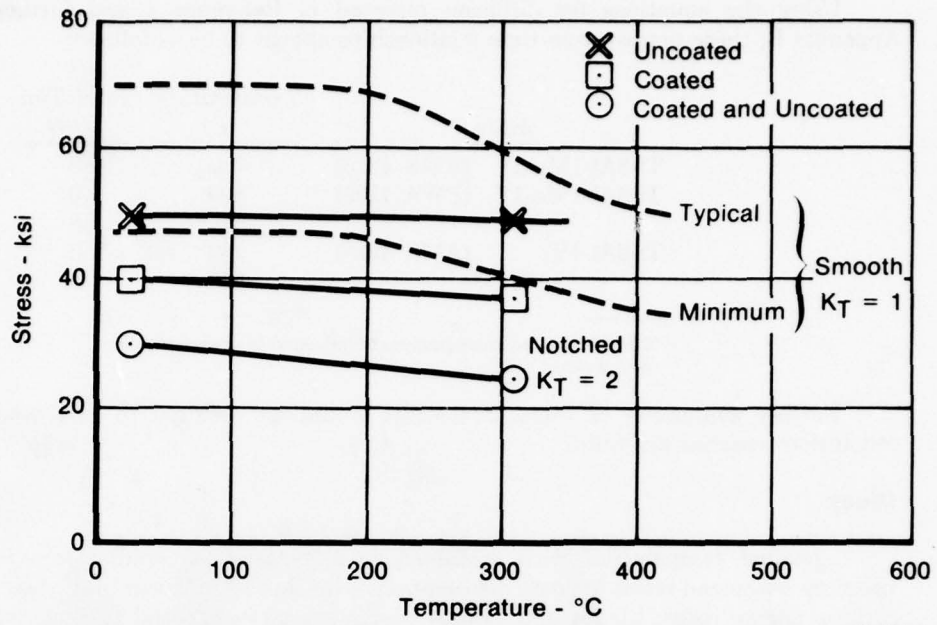


Figure 28. Ti 6Al-4V Fatigue Strength 10^7 Cycles

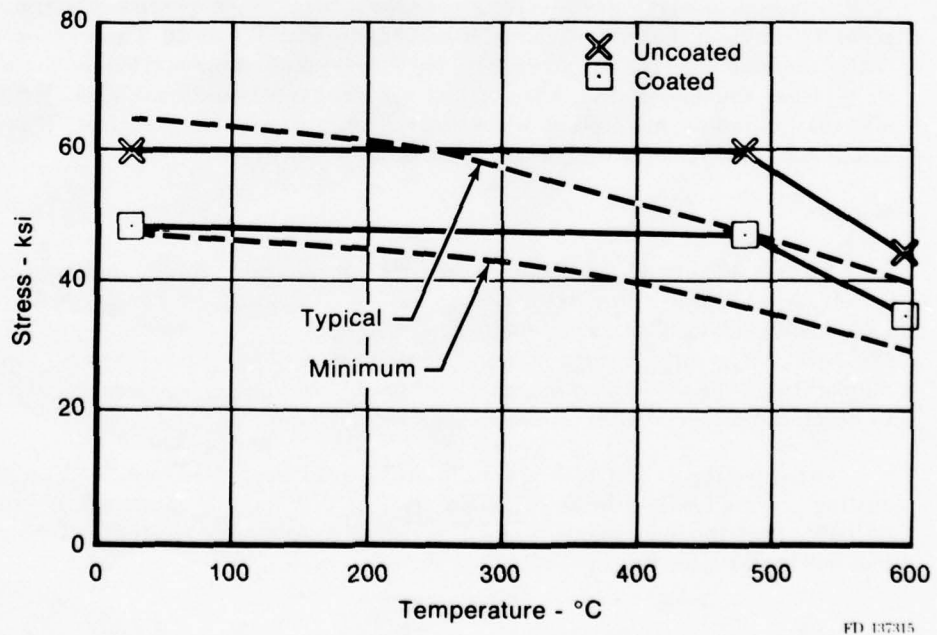


Figure 29. Ti 8Al-1Mo-1V Smooth Fatigue Strength 10^7 Cycles

Using the equations for diffusion reported in Reference 1 and further described in Appendix B, these temperature-time relationships appear to be as follows:

<u>Alloy</u>	<u>Temperature (°C)</u>	<u>Hold-Time (hr)</u>
Ti-8Al-1Mo-1V (AMS 4916)	760	3
Ti-8Al-1Mo-1V (PWA 1202)	890	0*
	575	8
Ti-6Al-4V (AMS 4928)	760	3
	575	3

*0 Hold time indicates heat-up to indicated temperature, then cool down.

Further evaluation of these treatments would be required to determine whether the conclusions reached are valid.

Creep

Elevated temperature with sustained load exposure of components fabricated from titanium alloys can result in creep deformation of the parts which can limit their useful life. Gas turbine engine disks, blades and exhaust ducts are often exposed to a combination of high temperatures and stresses. Components affected by this deformation can, over a period of time, change dimensions enough to prevent further use.

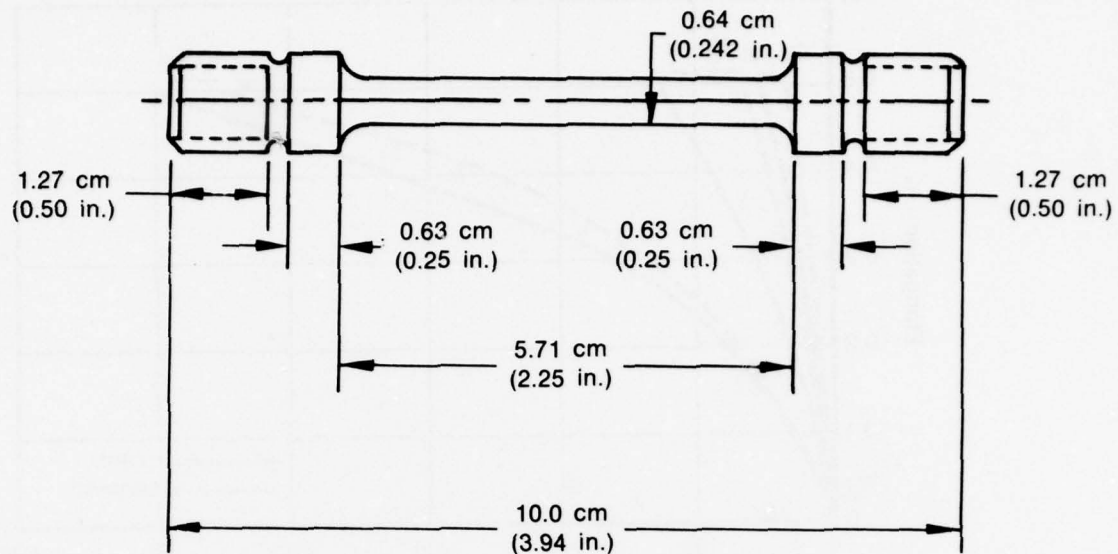
Test Procedure

Creep curves, graphically representing the relationship between creep strain and time, were generated using specimens machined in accordance with Figure 30. These specimens, in both the coated and uncoated condition, were heated to test temperature and loaded to the predetermined stress level calculated from the original specimen cross-sectional area. Extensometer holes allowed continuous monitoring of specimen length as a function of time. This data permitted calculation of percent creep and the plotting of creep curves.

Results

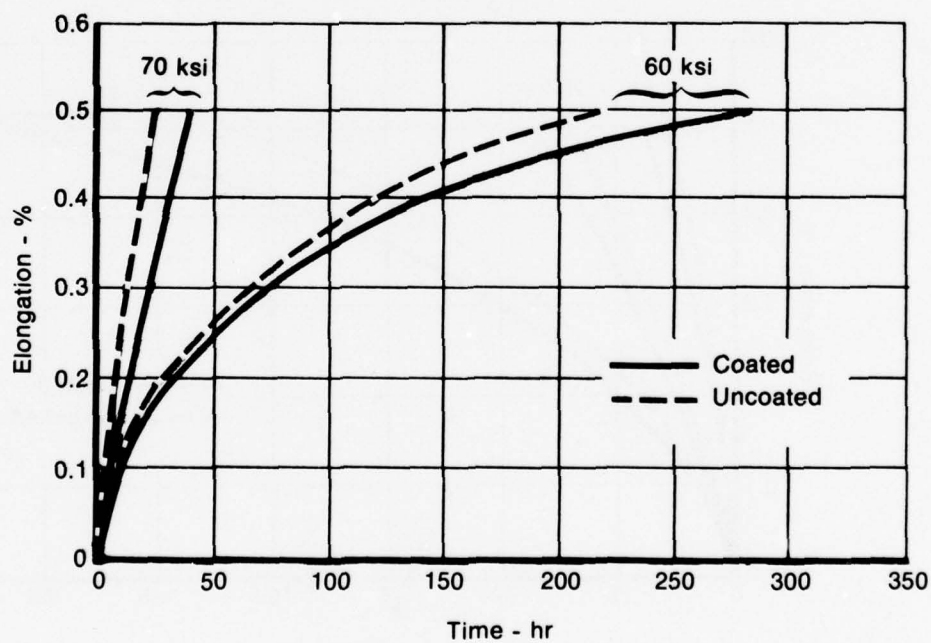
Figures 31 through 33 illustrate the creep results of this test program. The creep rate of Ti-8Al-1Mo-1V (fully heat-treated material) was reduced by coating with the chromium-molybdenum alloy. This corresponds to increasing the life of the titanium alloy components since the lower creep rate results in less deformation for any time interval. Depending on the temperature/time/stress level being tested from 10 to 60% increases in expected life were achieved by coating the specimens with chromium-molybdenum.

Other testing on Ti-6Al-2Sn-4Zr-2Mo (fully heat treated) showed the heat-treat cycle of the coating process resulted in an increased creep rate. Figure 34 shows that both the coated and uncoated heat-treated specimens had a creep rate considerably greater than the specimens which had not been exposed to the 760°C/3 hr diffusion cycle.



FD 137320

Figure 30. Creep Specimen



FD 137321

Figure 31. Creep Curve for Ti 8Al-1Mo-1V at 427°C (800°F)

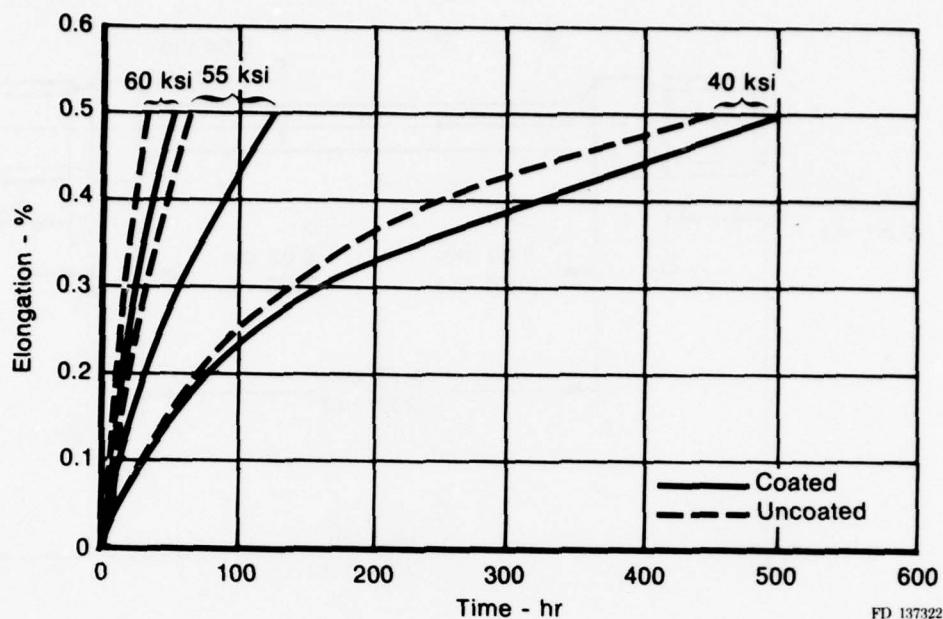


Figure 32. Creep Curve for Ti 8Al-1Mo-1V at 454°C (850°F)

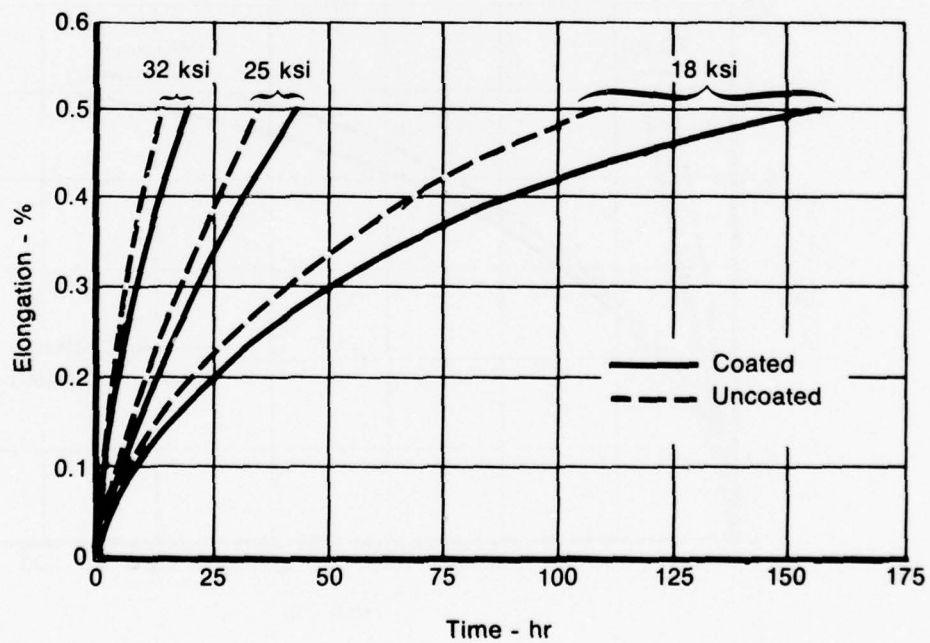


Figure 33. Creep Curve for Ti 8Al-1Mo-1V at 538°C (1000°F)

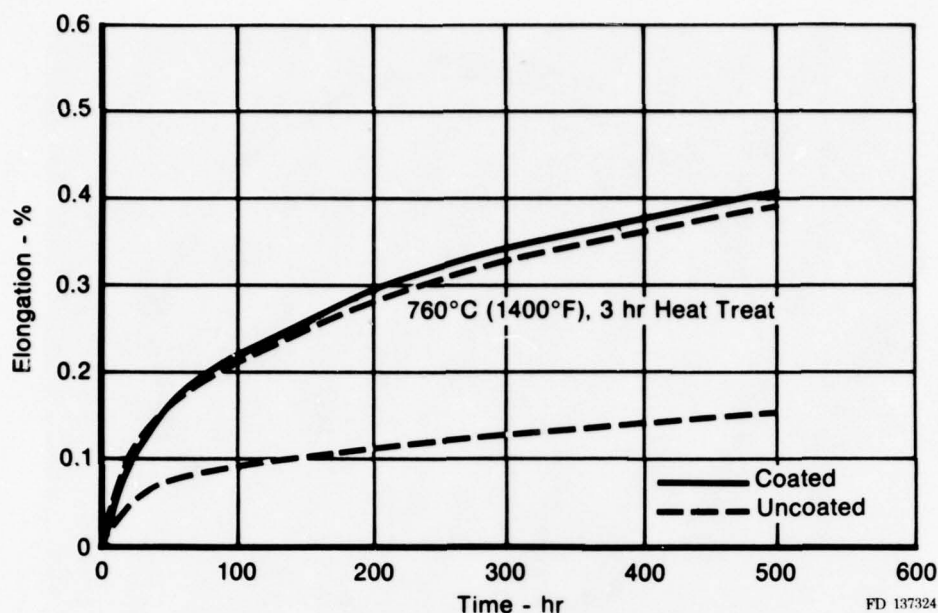


Figure 34. Creep Curves for Ti 6Al-2Sn-4Zr-2Mo at 482°C (900°F) With a Stress of 55 ksi

Discussion of Results

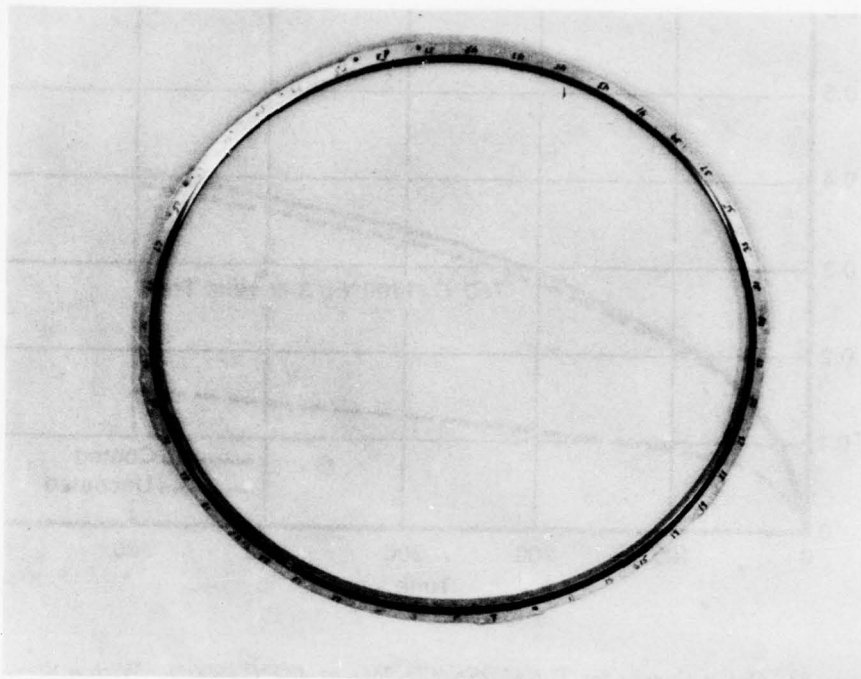
The creep rate of titanium alloys can be reduced by coating with chromium-molybdenum alloy. The affect is probably related to the reduced oxidation rate of the coated material which essentially protects the titanium from the oxygen of the environment. This type of protection has been reported in the past (Reference 7).

Coating of Ti-6Al-2Sn-4Zr-2Mo material for creep-limited components will require a modified heat-treat cycle for diffusion to prevent a degradation of properties.

SCALE-UP AND PLATING OF A COMPLEX GEOMETRY ENGINE PART

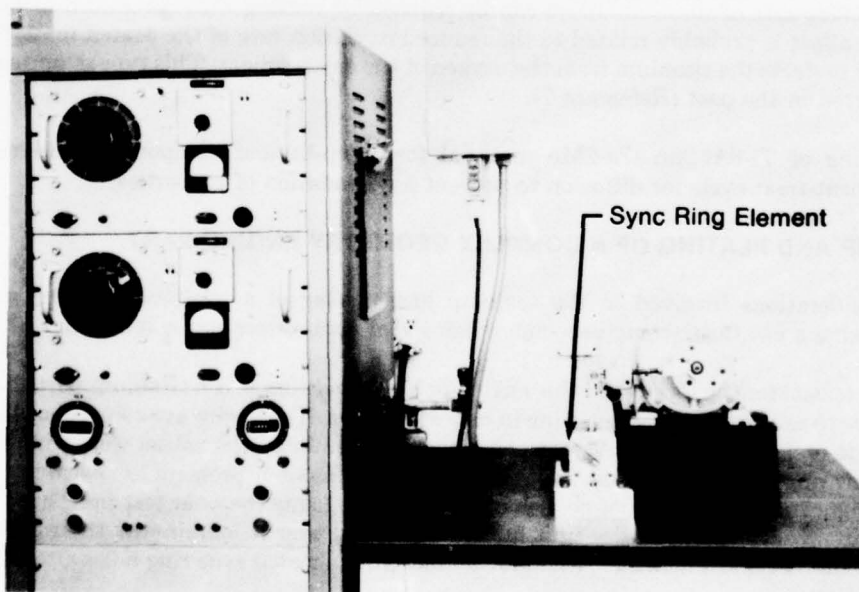
Considerations involved in the scale-up and plating of a complex geometry part were studied using a gas turbine engine synchronizing rear compressor stator inlet ring.

The reason for the interest in this engine part was because it is a titanium part that fails in service due to severe hole wear resulting in hole elongation. The engine sync ring, shown in Figure 35, is made of Ti 8Al-1Mo-1V. A wear test rig which simulated the actual wear conditions seen by the sync ring in the engine was used to characterize this wear problem as shown in Figures 36 and 37. Figure 38 shows a plot of hole elongation vs time using the wear test rig. The chromium-molybdenum coating, with a dry film lubricant applied over it, eliminated the wear problem. Figure 39 shows typical hole elongation for coated and uncoated sync ring holes after rig testing.



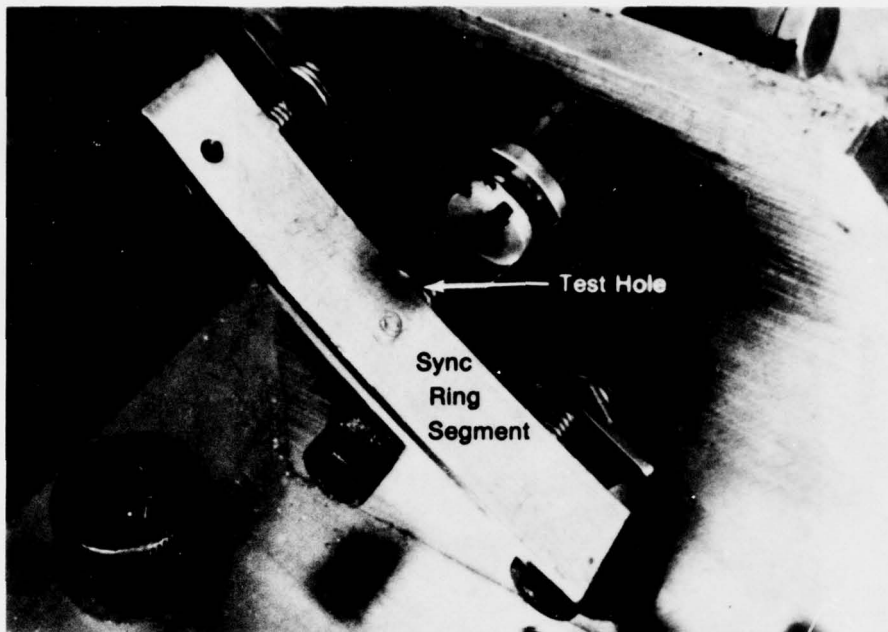
FD 161408

Figure 35. Titanium Alloy Gas Turbine Engine Sync Ring, 67.8 cm Dia



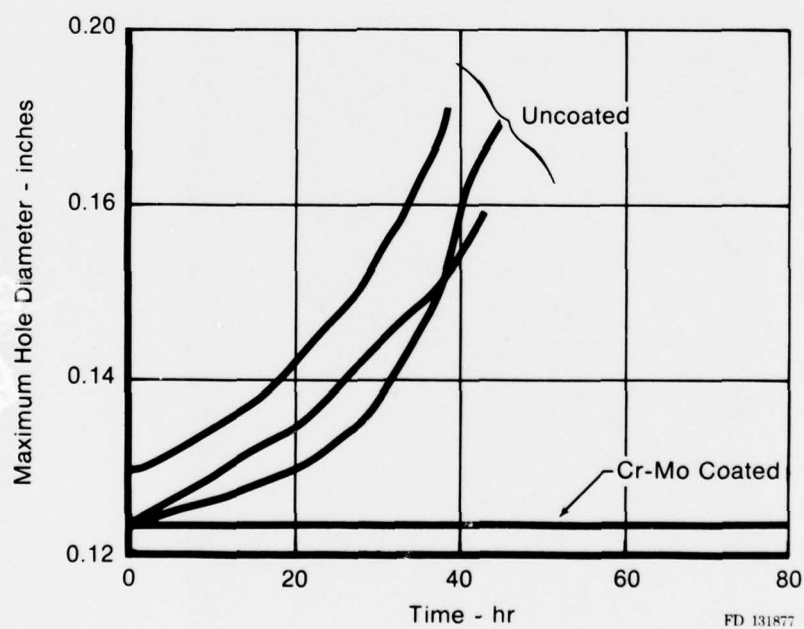
FD 137325

Figure 36. Rig Test Equipment



FD 137326

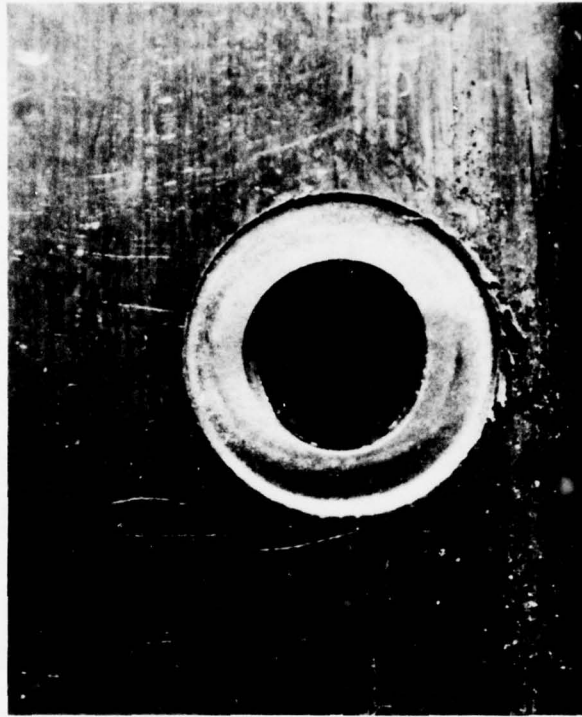
Figure 37. Rig Test Specimen



FD 131877

Figure 38. Accelerated Test Results

Uncoated



FD 131880

Cr-Mo Coated



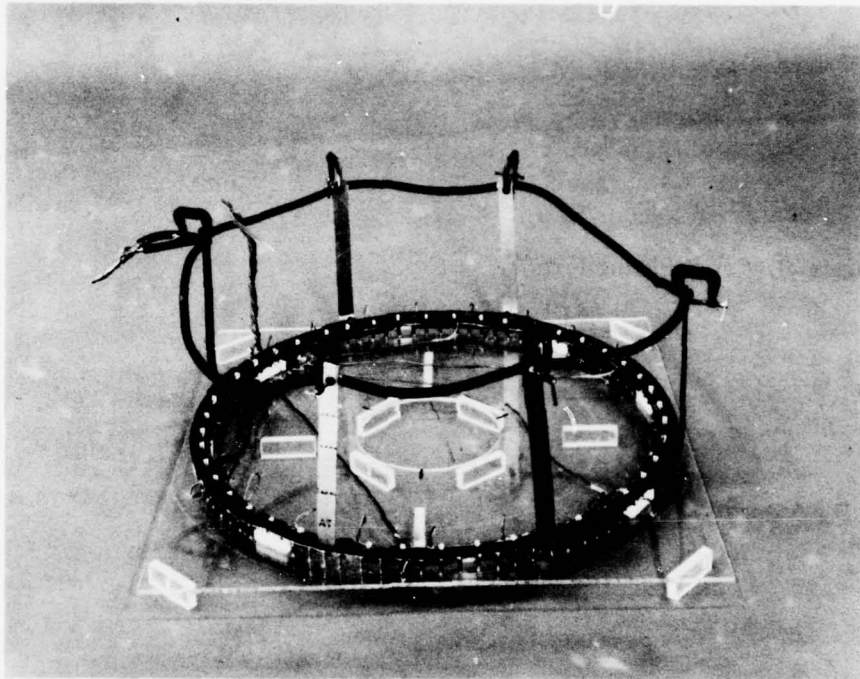
Mag: 6X

Figure 39. Engine Sync Ring Hole Appearance After Accelerated Wear Test

To plate an entire engine sync ring, due to the complex geometry and large numbers of holes requiring a protective coating, the complex fixture shown in Figure 40 was developed. This fixture consisted of a lead wire anode circling the ID and OD and supported by short lead rods affixed to a plexiglass plate (Figures 41 and 42). In Figure 42, the center lead anode lead wires can be seen with loops which are used to transport current from the ID lead anode to the center lead anode.

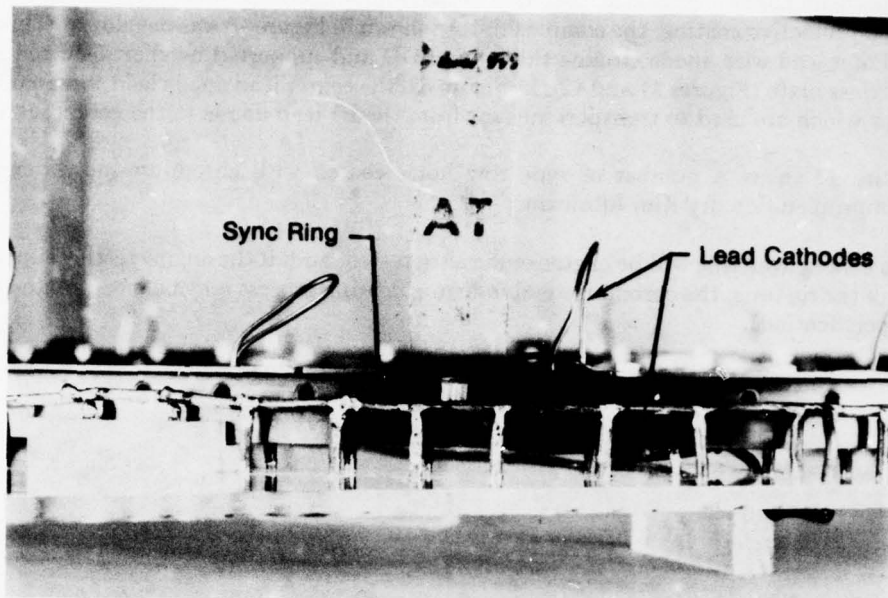
Figure 43 shows a number of sync ring holes coated with chromium-molybdenum and molybdenum disulfide dry film lubricant.

The coated sync ring will be engine endurance tested, and, if the engine testing supports the findings of the rig tests, the chromium-molybdenum coating process may be incorporated into the engine specifications.



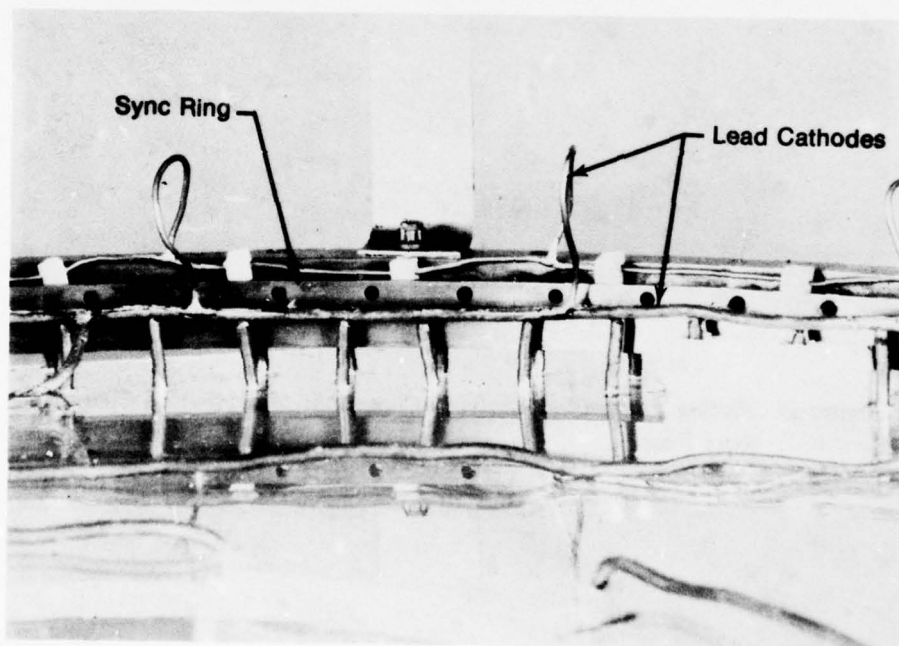
FE 161780

Figure 40. Plating Fixture for Applying Chromium-Molybdenum Coating to Sync Ring



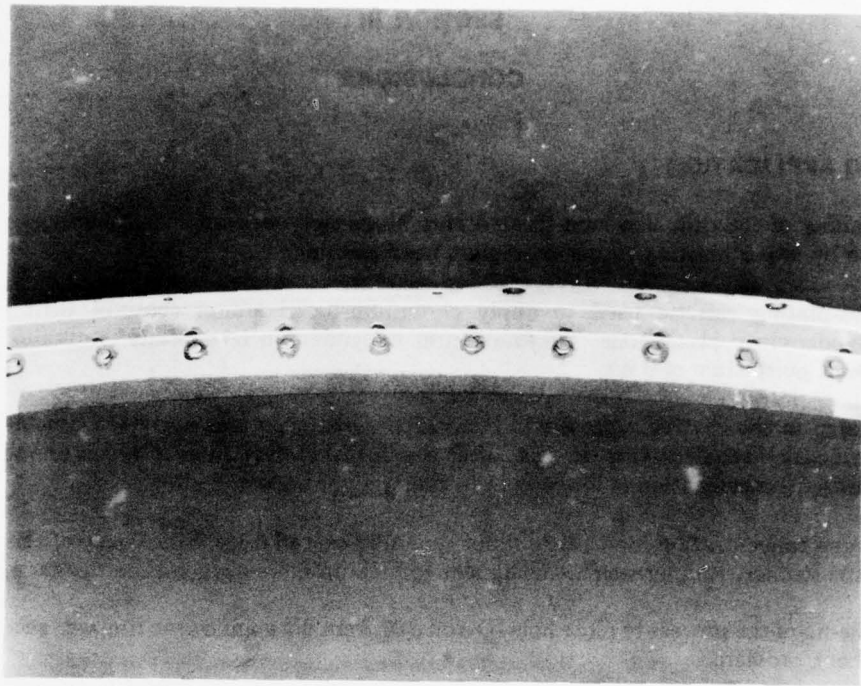
FD 137327

Figure 41. OD of Plating Fixture



FD 137328

Figure 42. ID of Plating Fixture



FE 162430

Figure 43. Chromium-Molybdenum Coated Sync Fixture

SECTION III

CONCLUSIONS

COATING APPLICATION

1. Cleaning of the diffusion heat-treated chromium-molybdenum coated surfaces should be done by vapor blasting rather than glass bead peening.
2. Pulse-plating can be used to apply chromium-molybdenum coatings with controlled molybdenum compositions. The maximum molybdenum in deposits is obtained using a current pulse-time of 3 ms.
3. No step in the coating process will increase the hydrogen content of the base alloy above specification requirements. In fact, after the diffusion heat treat in argon or vacuum, the hydrogen concentration is lower than in the original uncoated alloy.
4. Oxygen concentration increases did not generally exceed 0.1%. Since most of the oxygen is on the surface, thinner specimens appear to pick up more oxygen than thicker specimens.
5. Scale-up of the process to plate hole ID's on a 67.8 cm dia engine sync ring was accomplished without problem.
6. Pulse plating can be used to deposit a chromium-molybdenum coating with a gradient of hardness across its thickness.

COATING PROPERTIES

1. Pulse-plated deposits are generally softer and have better wear resistance than direct-current plated deposits.
2. Crack-free deposits can be obtained using either direct-current or pulse-current intervals of 40 ms or less.
3. Direct-current as-deposited coatings on titanium alloys had a hardness of 780 VHN while the pulse-plated deposits had a hardness of only 330 VHN. After heat treating above 650°C, the hardness decreased. The higher the temperature, the lower the hardness. The normal 760°C at 3 hr heat treat reduced the direct-current plated coating to 650 VHN and the pulse-plated coating to 291 VHN.
4. The wear resistance of direct-current plated chromium-molybdenum on steel is a factor of 3 better than conventional chrome plate on steel. Pulse-plating is a factor of 6 better than the conventional chrome.

COATING EFFECTS ON ALLOY PROPERTIES

1. The tensile strength, yield strength, and elongation properties of AMS 4911 remain unaffected by the processing steps required for chromium-molybdenum coating. Further, parts may be stripped of chromium-molybdenum and recoated without affecting these mechanical properties.

2. Although the coating did not reduce the fatigue strength of Ti-8Al-1Mo-1V, the diffusion heat treat did reduce the fatigue strength of fully heat-treated (solution + age) Ti-8Al-1Mo-1V. The diffusion heat-treat temperature and time must therefore be carefully selected to prevent this effect.
3. Coating increases the creep life of Ti-8Al-1Mo-1V by about 10 to 50% depending on the temperature-stress conditions.
4. High-temperature oxidation tests indicate the chromium-molybdenum coating will protect titanium from severe oxidation at temperatures up to 760°C. This elevates use temperatures for titanium alloys by at least 100°C (212°F).

SECTION IV

RECOMMENDATIONS

Based on the results of the first two phases of this study the following recommendations are made for continued development and evaluation of chromium-molybdenum coatings. This continuation will provide the next step in obtaining sufficient data on the coating system to allow wide use of chromium-molybdenum on aerospace system components.

1. Conduct a more extensive evaluation of both pulsed- and direct-current plated coatings on steel
2. Confirm the effect of diffusion heat-treat selection on maintaining fatigue properties of titanium alloys
3. Investigate effects of pulse-plating using different on-off pulse times on the deposit compositions, hardness, structure, and substrate adhesion
4. Continue wear tests to define the relationship of deposit compositions and hardness on the wear resistance
5. Apply the coating system to a variety of components for functional testing
6. Investigate changing the pulse parameters while plating to establish the effect of the gradient coating for wear resistance.
7. Prepare a suggested specification for procurement of current state-of-the-art chromium-molybdenum coatings on military hardware.

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APPENDIX A

CHROMIUM-MOLYBDENUM APPLICATION PROCEDURE FOR TITANIUM

1.0 SOLUTION MAKEUP

1.1 Self-Regulating Plating Bath

Chromic Acid ⁽¹⁾	40 oz/gal (300 g/l) 1% Molybdenum deposit - 10 oz/gal (75 g/l)
Ammonium Molybdate (NH ₄) Mo ₇ O ₂₄ × H ₂ O	3% Molybdenum deposit - 12.6 oz/gal (95 g/l)

1.1.1 Dissolve the chromic acid in deionized water at room temperature.

1.1.2 Dissolve the ammonium molybdate solution in deionized water at 60°C ± 5°C (140°F ± 10°F).

1.1.3 Add the ammonium molybdate solution to the chromic acid and adjust volume to operating level.

1.1.4 Adjust temperature of plating solution to 38°C ± 2°C (110°F ± 5°F).

1.2 Vapor Blast

Novaculite 200 ⁽²⁾	480-720 g/l (4 to 6 lb/gal)
Antisolidifying Compound ⁽³⁾	113 g/22.7 Kg (4 oz/50 lb) abrasive
Corrosion Inhibitor ⁽⁴⁾	4 ml/l (15 ml/gal)

1.2.1 Fill tank to about ¾ of operating level with water.

1.2.2 Add abrasive slowly while circulating pump is on.

1.2.3 Add antisolidifying compound.

1.2.4 Add corrosion inhibitor.

1.2.5 Fill to operating level.

1.3 Etch Solution

Hydrofluoric Acid (48%)	2.1 ml/l (8 ml/gal)
Sodium Chromate	20 g/l

1.3.1 Dissolve the required amount of sodium chromate in deionized water.

1.3.2 Add required amount of hydrofluoric acid.

2.0 PROCEDURE FOR DIRECT CURRENT PLATING

- 2.1 Mask area not to be plated.
- 2.2 Vapor blast surface to be plated with wet abrasive at 0.4 to 0.7 MPa (50 to 100 psi).
- 2.3 Rinse thoroughly in clean running water.
 - 2.3.1 Cleaned surface must not be allowed to dry prior to plating. Submerge part in deionized water.
- 2.4 Etch for 5 to 10 sec to produce chrome conversion coating.
- 2.5 Using lead anodes, immerse part in plating solution with current "off."
- 2.6 Turn on power supply and raise current to 11.7 ASD (0.75 ASI) for 2.5 min.
- 2.7 Plate at 46.7 ASD (3.0 ASI) for 30 min.
- 2.8 Rinse thoroughly in deionized water.
- 2.9 Heat treat in vacuum at 760°C for 3 hr.
 - 2.9.1 (Option) — Vapor blast surfaces.

3.0 PROCEDURE FOR PULSE PLATING

- 3.1 Mask area not to be plated.
- 3.2 Vapor blast surface to be plated with wet abrasive at 0.4 to 0.7 MPa (50 to 100 psi).
- 3.3 Rinse thoroughly in clean running water.
 - 3.3.1 Cleaned surface must not be allowed to dry prior to plating. Submerge part in deionized water.
- 3.4 Etch for 5 to 10 sec to produce chrome conversion coating.
- 3.5 Using lead anodes, immerse part in plating solution with current "off."
- 3.6 Turn on power supply and raise current to 11.7 ASD (0.75 ASI) for 2.5 min.
- 3.7 Plate at 46.7 ASD (3.0 ASI) for 30 min with the pulse plater set at 3 ms "on" and 3 ms "off."
- 3.8 Rinse thoroughly in deionized water.
- 3.9 Heat treat in vacuum at 760°C for 3 hr.
 - 3.9.1 (Option) — Vapor blast surfaces.

⁽¹⁾United Chromium, SRHS CR 110

⁽²⁾Vapor Blasting Mfg Co., NVB No. 200

⁽³⁾Vapor Blast Mfg Co., No-Pak

⁽⁴⁾Reilly-Whiteman-Walton Co., X61-60 water conditioner

APPENDIX B

CALCULATION OF DESIRABLE TIME-TEMPERATURE DIFFUSION CYCLE

1.0 REQUIREMENT 1

1.1 Diffusion heat-treat should be compatible with previous heat-treat history.

1.2 Example: Fully heat-treated Ti-8-1-1 has been:

1.2.1 Solutioned at 885 to 1025°C for 1 hr.

1.2.2 Stabilized at 550 to 600°C for 8 hr.

Therefore, diffusion heat-treat should be either long time at 550 to 600°C or short time at 885 to 1025°C followed by 8 hr at 550 to 600°C.

2.0 REQUIREMENT 2

2.1 To get suitable adhesion, depth of diffusion should be at least as great as depth obtained by heat treating at 760°C for 3 hr.

2.2 $X = 4 [tD \exp(-Q/RT)]^{1/2}$ setting X_1 (depth obtained at 760°C for 3 hr) = X_2 (depth obtained for some other time-temperature combination), and solving for t (time required to get desired depth of diffusion at temperature T_2) results in:

$$t_2 = t_1 \exp \left(Q \frac{(T_1 - T_2)}{RT_1 T_2} \right)$$

3.0 FOR REQUIREMENT 1 TO BE MET:

3.1 A temperature must be selected from the ranges of 885 to 1025°C or 550 to 600°C.

3.2.1 Assuming the lower temperature of 600°C is used then:

$$t_2 = 3 \exp \left[\frac{75,100 (1033 - 1198)}{1.98 (1033) (873)} \right] = 2510 \text{ hr}$$

3.2.2 A heat-treat time of 2510 hr (15 weeks) would be required if the lower temperature were used, this is not practical.

3.2.3 Recalculating the time based on a diffusion temperature of 925°C:

$$t_2 = 3 \exp \frac{75,100 (1033 - 1198)}{1.98 (1033) (873)} = 0.019 \text{ hr}$$

3.2.4 A heat-treat time of 0.019 hr or 1.2 min at 925°C is required to get the same diffusion depth produced at 760°C for 3 hr. This temperature exposure will resolution the alloy and must be followed by an 8-hr, 575°C stabilization heat treat.

APPENDIX C

CHROMIUM-MOLYBDENUM APPLICATION PROCEDURE FOR STEELS

1.0 SOLUTION MAKEUP

1.1 Self-Regulating Plating Bath

Chromic Acid ⁽¹⁾	40 oz/gal (300 g/l) 1% Molybdenum deposit - 10 oz/gal (75 g/l)
Ammonium Molybdate (NH ₄) Mo ₇ O ₂₄ × H ₂ O	3% Molybdenum deposit - 12.6 oz/gal (95 g/l)

- 1.1.1 Dissolve the chromic acid in deionized water at room temperature.
- 1.1.2 Dissolve the ammonium molybdate solution in deionized water at 60°C ± 5°C (140°F ± 10°F).
- 1.1.3 Add the ammonium molybdate solution to the chromic and adjust volume to operating level.
- 1.1.4 Adjust temperature of plating solution to 38°C ± 2°C (110°F ± 5°F).

2.0 STRESS RELIEF TREATMENT

- 2.1 All steel parts having an ultimate tensile strength of 150,000 psi (1034 MPa) and above, which are machined, ground, cold formed or cold straightened, shall be baked at a minimum of 191 ± 14°C (375 ± 25°F) for 3 hr or more prior to cleaning and plating for the relief of damaging residual tensile stresses.

3.0 PROCEDURE FOR DIRECT-CURRENT PLATING

- 3.1 Mask area not to be plated.
- 3.2 Anodic etch at 1 to 3 amps in.² for ½ to 10 min. Etch in the following solution (preferred) or in the plating bath, 1.1:

Chromic Acid	30 to 40 oz/gal
Temperature	90 to 130°F

Note: Etching increases iron contamination in the plating bath, therefore the use of solution 3.2 is recommended.

- 3.3 Using lead anodes, immerse part in plating solution with current "off."
- 3.4 Turn on power supply and raise current to 11.7 ASD (0.75 ASI) for 2.5 min.
- 3.5 Plate at 46.7 ASD (3.0 ASI) for 30 min.
- 3.6 Rinse thoroughly in clean running water.

3.7 Embrittlement relief.

- 3.7.1 All coated steel parts having a hardness of Rockwell C40 and higher shall be baked at a minimum of $191 \pm 14^{\circ}\text{C}$ ($375 \pm 25^{\circ}\text{F}$) for 3 hr or more, within 4 hr after plating, to provide hydrogen embrittlement relief.

4.0 PROCEDURE FOR PULSE-CURRENT PLATING

4.1 Mask area not to be plated.

- 4.2 Anodic etch at 1 to 3 amps in.² for $\frac{1}{2}$ to 10 min. Etch in the following solution (preferred) or in the plating bath, 1.1:

Chromic Acid	30 to 40 oz/gal
Temperature	90 to 130°F

Note: Etching increases iron contamination in the plating bath, therefore the use of solution 4.2 is recommended.

- 4.3 Using lead anodes, immerse part in plating solution with current "off."
- 4.4 Turn on power supply and raise current to 11.7 ASD (0.75 ASI) for 2.5 min.
- 4.5 Plate at 46.7 ASD (3.0 ASI) for 30 min with the pulse plater set at 3 ms "on" and 3 ms "off."
- 4.6 Rinse thoroughly in deionized water.
- 4.7 Embrittlement relief.

- 4.7.1 All coated steel parts having a hardness of Rockwell C40 and higher shall be baked at a minimum of $191 \pm 14^{\circ}\text{C}$ ($375 \pm 25^{\circ}\text{F}$) for 3 hr or more, within 4 hr after plating to provide hydrogen embrittlement relief.

⁽¹⁾United Chromium, SRHS CR 110

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